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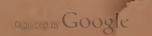
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### CHEMICAL ABSTRACTS

Vol. 5. OCTOBER 10, 1911.

No. 19

#### 1. APPARATUS.

L. C. JONES.

A New Lecture Apparatus. FRITZ CASPARI. Chem. Zig., 35, 183.—The app. is designed to be used as a lecture app. to show how Zn when chem. pure is not much

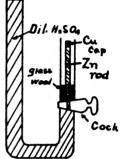
acted upon by pure dil. H<sub>2</sub>SO<sub>4</sub>; but when an impurity (Cu cap) is in contact with the metal, rapid action takes place. A study of the figure will make its construction and use clear.

J. R. WITHROW.

A New Distillation Apparatus for Nitrogen Determinations. NEUBAUER. Landw. Vers. Sta., 74, 376-80.

E. C. LATHROP.

Electrically Controlled Constant Temperature Water Bath. H. C. Gorr. J. Ind. Eng. Chem., 3, 506-7; Bur. Chem., Circ. 72.—A device by which water is added to a bath automatically. By variations of a mercurial thermostat a circuit is made or broken thru an ordinary sounder. The pressure of the sounder's electromagnets upon the water supply rubber tube, controls the water in the bath. Illustrated. C. A. C.



Two Simple Forms of Gas-pressure Regulators. E. STANSFIELD. Chem. News, 104, 3-4.—One cylindrical vessel open at the top and bottom so that water passes freely in or out, is placed within a second. The gas comes into the inner cylinder and at low pressure a float carrying a gas rod opens the exit tube, but at higher pressures the water level is depressed, the float falls and cuts down the capacity of the gas delivery tube.

W. H. McLauchlan.

Automatic Gas Analysis for Boiler House and Chemical Works Control. Anon. Met. Chem. Eng., 9, 327.—A general description of the Simmance Abady type of automatic gas analyzers, with cut and diagram of the CO<sub>2</sub> machine and cut of recorder for corrosive gases.

E. C. S.

Detection of Inflammable Gases in Air. A. Phil. IP AND L. G. Steel. J. Gas Lighting, 115, 233.—A new app. that embodies a device for guarding against the overheating of Pt. The app. acts automatically, depending upon the sensitiveness of a relay, and gives a warning signal by closing an elec. circuit and ringing a bell when the proportion of gas or vapors amts. to 1/4 of that which corresponds to the lower explosive limit of that particular gas or vapor and air.

C. A. C.

An Improved Funnel. P. BLACKMAN. Chem. News, 104, 30.—The funnel is made of glass and of various sizes but the last third of the cone shape is replaced by a cylinder which has the usual dropping tube. The advantages are quicker filtration, as much of the paper is not in contact with the glass.

W. H. McL.

Platinum Laboratory Utensils. PERCY H. WALKER AND F. W. SMITHER. Bur. Chem., Bull. 137, 180.—The article calls attention to the fact that a great deal of Pt ware, such as crucibles, dishes, etc., offered for sale at the present time is of inferior quality, some of it being absolutely unfit for use in a laboratory. Methods of testing

Pt laboratory app. are described, and suggestions for a standard specification for such ware are given.

PERCY H. WALKER.

Funnel Support. P. BLACKMAN. Chem. News, 104, 30-1.—This is a glass disk, fluted in concentric rings and having at the center a tube, 0.5 in. in diam. and 1.5 in. high, each end being funnel-shaped, in which the funnel is placed. The holder is placed on a beaker and by inverting it, the funnel can be placed in a high or low position.

W. H. McL.

An Improved Pycnometer for Glycerol. L. W. BOSART, JR. J. Ind. Eng. Chem., 3, 508.—The improvement consists of a small cylindrical funnel 38 × 15 mm. ground to fit the capillary, in place of the usual cap. Glycerol poured into the pycnometer may be weighed at any time, since the funnel will accommodate any expansion. Illustrated.

New Form of Direct Reading Candlepower Scale and Recording Device for Precision Photometers. G. W. MIDDLEKAUFF. Bull. Bur. Standards, 7, 12-43.—The detailed description of this instrument is too long and complicated for a brief abstract. Some of the claims advanced are given here. The direct reading scale and automatic recorder were designed primarily for precise, photometric work in the Bureau of Standards but have been adapted for use with ordinary commercial photometers. The adaptation combines accuracy with the direct reading feature, thus eliminating labor and possible errors of reading and recording; all tedious computations are done away with. The work of reading and recording is rapid and accurate. There is no strain on the observer's eyes, and no complex adjustments are required. The record is simple, permanent and complete.

Modified Colorimeter and some Tests of its Accuracy. E. D. CAMPBELL AND W. B. HURLEY. J. Am. Chem. Soc., 33, 1112-5.—Two half-mirrors are permanently fixed at an angle of 45° above the tubes for color comparisons. By means of a plunger, the column in the known tube may be varied to compare with unknown. An adjustable mirror at the bottom of the two columns serves to reflect the light thru the columns, which is in turn transmitted to the eye by the half-mirrors above the tubes. C. A. C.

A Photometric Attachment for Spectroscopes. P. G. NUTTING. Bull. Bur. Standards, 7, 239-41.—The instrument attains high sensibility, economy of light, and variable dispersion. By means of 2 reflecting prisms, 2 nicols and a lens, a real image of the photometric surface is thrown on the spectroscope slit by an achromatic lens, and may thus be brought into the plane of the slit. The 2 beams of light to be compared are brought to equality by rotating one of the nicols. The photometric surface has alternate and silvered strips. Sensibilities varying from 0.015-0.018 were recorded, and corrections were very small.

M. Heidelberger.

Strongly Dispersive Monochromator and Spectrum Apparatus for Strong Light. H. DU BOIS AND G. J. ELIAS. Berlin and Haarlem. Z. Instrument., 31, 79-87.—Besides the characteristics given in the title, this app. was to satisfy requirements of magneto-optics and be available for direct vision as well as photographic work. The details of construction are too numerous to be abstracted briefly. The app. is now being mfg. by Fr. Schmidt & Haensch, Berlin.

E. J. WITZEMANN.

A Toepfer Measuring Apparatus for Spectrographs. F. Goos. Z. Instrument., 31, 52-5.—An illustrated description of this app. together with exhaustive tests of the accuracy of the screw thread on which the readings depend. This screw proved to be unusually good. App. mfd. by O. Toepfer & Sohn, Potsdam.

E. J. W.

The Use of Cylindrical Lenses in Spectrum Apparatus. E. Gehrcke. Phys.-Tech. Reichsanstalt., Charlottenburg. Z. Instrument., 31, 87-9.—In most app. the wave length is detd. by measuring so-called "lines" or narrow strips of light. If all

the light so distributed in a line were concentrated in a small spot observations would often be easier and photographs more quickly obtained. These lines may be reduced to spots by placing a cylindrical lense at the right distance in front of the picture with its axis perpendicular to the spectral line. Some photographs are shown. A small stirring rod (5 mm.) may even be used sometimes.

E. J. WITZEMANN.

Activity of the Physikalisch-Technischen Reichsanstalt in 1910. Anon. Z. Instrument., 31, 112-25, 151-63, 183-97.—A record of work in progress and finished, and citations to work published.

E. J. WITZEMANN.

Thermostat for Controlling the Temperature of a Room. EARL OF BERKELEY AND C. V. BURTON. *Phil. Mag.*, 21, 211-4.—A small interferometer room had its temp. controlled by elec. currents. A detailed description with dimensions of all working parts is given. On a trial, extending from 9.00 A.M. to 12.15 P.M. the temp. changed only  $\frac{1}{100}$  of a degree from the mean.

ROBT. F. EARHART.

A Replacable Unit System of Electric Heating Apparatus. M. M. Kohn. Trans. Am. Electrochem. Soc., 19, 299-310.—The author comments upon the impracticability of replacing the resistors in the common class of heaters, and describes a system of heaters with the heating unit placed in a structure separate and distinct from the chamber to be heated. The following advantages are claimed: Flexibility of construction, ease and rapidity of replacement of damaged part, very high efficiency, low maintenance cost, general adaptability to any and all forms of elec. heating app. Illustrations are shown. In the discussion of the paper, J. W. Richards pointed out that lab. resistance furnaces when painted white on the outside, using Al paint, consumed considerably less power than when the covering was black or rusty.

WM. VAN KEUREN.

Simple Apparatus for Recovery of Solvent in Analytical Fat Extractions. F. Voll-RATH. Seifensieder Ztg., 38, 814-6.—The app. which is suitable for solvents heavier than water, consists of a small flask with wide mouth having a 2-holed cork. Thru one hole an extraction tube 35 mm. wide and 200 mm. long and ending in a drawn-out narrow tube is inserted. Thru the other hole a glass tube passes to a condenser bottle having a cord with 2 holes, thru 1 of which a long glass tube passes to act as condenser. During the extraction this app, is disconnected from the extractor. The extraction tube contains the extraction thimble which rests upon a short glass funnel. A reflux condenser is attached to the extractor; and the extraction flask is heated in an oil bath. When the extraction is complete the reflux condenser is disconnected from the extraction tube and a small flask containing H<sub>2</sub>O for generating steam is connected and at the same time the condenser bottle is connected with the extraction flask and as much solvent distilled over as possible when steam is introduced into the extraction tube and the solvent removed from the thimble. The flame is not removed from extraction flask during this operation. When all the solvent is removed by the steam, a current of air is E. SCHERUBEL. passed thru to remove the moisture.

Porcelain Funnels with a Fiftering Paste. F. Grenet and P. Boulanger. Compt. rend., 152, 1759-60.—The authors have prepared 2 forms of filter funnels both of which are especially adapted to laboratory work. Solns. containing finely divided ppts. such as BaSO<sub>4</sub>, CaC<sub>2</sub>O<sub>4</sub>, ctc., also viscous liquids as serums may be readily filtered.

W. F. Langelier.

Auto-acetinometer (Gravier). 5. New Official Bavarian Saccharimeter (WIRTH). 16.

GEHRCKE, E.: Die Anwendung der Zylinderlinse in Spectralapparaten. Berlin: J. Springer.



#### 2. GENERAL AND PHYSICAL CHEMISTRY.

JOHN JOHNSTON.

Atomic Heats of the Elements. J. KOENIGSBERGER. Z. Elektrockem., 17, 289-93, 407. C. G. F.

Recent Advances in High-temperature Gas Thermometry. A. L. DAY. Chem. News, 104, 51-3, 62-4.

J. J. M.

Significance of the Periodic Law. Fernando Sanford. Stanford Univ. J. Am. Chem. Soc., 33, 1349-53; cf. C. A., 5, 2585.—The difficulty that the at. wts. of successive elements do not increase by 4 or a multiple of 4 (the at. wt. of He or au  $\alpha$ -particle) is largely overcome by assuming 2 parallel lines of development, one starting from He the other from Li; the first series has even valencies, the second series has odd valencies. The significance of this scheme is briefly discussed.

J. Johnston.

Some Arbitrary Distinctions which are Made in Theoretical Chemistry. GIOVANNI MALFITANO. Pasteur Inst., Paris. van Bemmelen Gedenkboek, 368-79.—The author points out and discusses the arbitrary nature of the distinction between physical and general chem., between physical and chem. phenomena, between substances of definit and those of variable comp. (compds. and solns.), between "true" compds. and mol. complexes, and between absorption and chem. combination.

J. JOHNSTON.

Note on the Internal Pressure of a Liquid. W. C. McC. Lewis. *Phil. Mag.*, 21, 193-8.—The constant  $a/V^2$  in van der Waals' equation is discussed. The work of Dupré, Traube, Richards and others is compared. Robt. F. Earhart.

Relation of Temperature and Molecular Attraction. J. E. MILLS. Phil. Mag., 21, 84-113.—The first part of this paper is a reply to R. D. Kleeman (C. A., 5, 11, 13). In part two it is shown that a relation proposed by Dieterici connecting the d. of a liquid and its satd. vapor at the temp. of vaporization does not apply at low vapor pressures, whereas at higher pressures the agreement is excellent. In part three the author presents evidence from which he concludes: Change of temp. does not change the nature or the amt. of the mol. attractive force; the temp. merely dets. the orbit that the mol. will follow in obedience to the attractive force. ROBT. F. EARHART.

The Attainment of Temperatures Considerably below the Boiling Point of Helium. H. Kamerlingh Onnes. Leiden. van Bemmelen Gedenkboek, 441-6.—The lowest temp. hitherto attained was 2° abs. in 1909, by the evapn. of liquid He at a pressure of about 2 mm. In a specially constructed app., in which the He to be evapd. was contained in a vessel surrounded by liquid He, the author was able to evap. He at a pressure of about 0.2 mm. This corresponds, according to calc. by means of the van der Waals' formula, to an abs. temp. below 1.9°, and probably about 1.8°.

J. JOHNSTON.

Weight of a Falling Drop and the Laws of Tate. IX. The Drop Weights of the Associated Liquids, Water, Ethyl Alcohol, Methyl Alcohol and Acetic Acid; and the Surface Tensions and Capillary Constants Calculated from them. J. L. R. MORGAN AND A. McD. McAfer. Columbia Univ. J. Am. Chem. Soc., 33, 1275-90.—From detns. at various temps. the authors obtain the following formulas, which give the surface tension  $\gamma_t$  (expressed in dynes per cm.) at  $t^o$ : for  $H_1O$ ,  $\gamma_t = 75.872 - 0.1547t - 0.000222t^2$ ; MeOH,  $\gamma_t = 23.740$  (1 - 0.003514t); EtOH,  $\gamma_t = 23.295$  (1 - 0.00341t); HOAc,  $\gamma_t = 28.749$  (1 - 0.00325t). Analogous equations are given for the capillary const. The measurements indicate that each of the above liquids is associated, and that the results obtained by this method are more consistent than those obtained by other methods heretofore.

Some Physical Chemistry Measurements. ADOLFO P. CASTANARES. México.

Anales inst. med. nacional, 11, 75-6.—The following b. ps. were detd. as part of a plan for the more exact detn. of some physical constants. Many of the substances were prepared synthetically and all were highly purified. Temps. are corrected in all cases and the pressure reduced to 0°. EtOH, 71.9°588; MeOH, 58.2°587-9; CHCl<sub>3</sub>, 53.35°587-5; Et<sub>2</sub>O, 27.6°588-9; acetone, 48.75°587-9; C<sub>4</sub>H<sub>6</sub>, 72.0°588; toluene, 101.55°588; CCl<sub>4</sub>, 68.2°583-3; CS<sub>3</sub>, 38.6°591; AcOH, 109.5°587; MeI, 34.6°587; aniline, 174.4°589-4; nitrobenzene, 199.8°587-9; benzoyl chloride, 186.2°587-3.

H. S. Paine.

Molecular Movement in Viscous Liquids. Jean Perrin and Niels Bjerrum. Compt. rend., 152, 1569–71.—The authors succeeded in measuring the Brownian movement in emulsions of gamboge in 88% glycerol, the viscosity of which is about 115 times as great as that of water. The methods employed were those previously used by Perrin with aq. solns. (tho in the present case the particles tend to conc. in the upper layers of the soln.). The values of N obtained vary from  $64-74 \times 10^{22}$  (most probable value of N is  $68.5 \times 10^{22}$ ).

Orientation of Fluid Crystals by the Magnetic Field. C. MAUGUIN. Compt. rend., 152, 1680-4.—From expts. with azoxyphenetole and azoxyanisole, the author concludes that, under the action of a magnetic field, such liquids assume all the optical properties of a uniaxial cryst. plate, the optic axis of which is in the direction of the lines of force, and parallel to the surfaces of the slide and cover glass; without the magnetic field it behaves as if the optic axis were in a direction perpendicular to the glass surfaces.

J. JOHNSTON.

Weak Electrolytes and a Dynamical Theory of Solutions. Wm. SUTHERLAND. Phil. Mag., 21, 17-66.—The author extends his application of molecular potential energy and presents analytical relations derived with the aid of hypotheses concerning the nature and motion of mols. and the intermolecular forces. Detailed examples of the application of these relations are applied to the d. of solns. of EtOH and HOAc in H<sub>2</sub>O and the contraction which occurs on their formation. Similar results for the other chief physical properties of mixtures of H<sub>2</sub>O with EtOH and with the first four of the series of fatty acids—namely mol. refraction and dielect. capacity, viscosity, sp. heat, heat of mixing, and surface energy—are tabulated and discussed.

ROBT. F. EARHART.

Mechanical Stimulus to Crystallization. II. S. W. Young and R. J. Cross. Stanford Univ. J. Am. Chem. Soc., 33, 1375-88; cf. C. A., 5, 1009.—A series of expts. on the influence of the mass and number of foreign particles and of variation in viscosity of the liquid upon the degree of supercooling of aq. solns. yielded results in harmony with the assumption that the effect of foreign particles is purely kinetic; or in other words, that their activity in stimulating crystn. is due to the occurrence of impacts between them.

J. Johnston.

The Dissolved Molecule and the Hypothesis of van't Hoff. ALBERT COLSON. Bull. soc. chim., 9, 576-83.—According to the author, it is inadmissible that HOAc, which in the vapor state exists as double mols. below about 180°, should exist in soln. at ordinary temp. as single mols. We must therefore conclude that the usually accepted cryoscopic constant for aq. solns. must be doubled so that it may refer to 1 mol.; and if we do this the mol. wt. of HCl (calc. from f. p.) is normal and the hypothesis of ions is unnecessary. This reasoning is supported by a few expts. on the f. p. and cond. of solns. of enlfates, especially the complex Cr sulfates of Recoura (cf. C. A., 2, 222, 1244).

Influence of Centrifugal Force upon Solutions. A. Dumanskii. Kiev. van Bemmelen Gedenkboek, 421-2.—Colloidal solns, centrifuged at 4000 r. p. in. gave ppts. in periods varying from a few mins. to a few hrs. Aq. solns. of salts (KCl, KBr,

UO<sub>2</sub>(NO<sub>2</sub>)<sub>2</sub>, Ba(NO<sub>2</sub>)<sub>2</sub>) when similarly treated show increased conc. at the periphery; this was detd. by measurements of the cond. of the inner and outer layers. Thus the behavior of colloidal solns. when centrifuged differs only in degree from that of salt solns.

J. JOHNSTON.

A Study of the Conductivity and Dissociation of Organic Acids in Aqueous Solution between 0° and 35°. E. P. WIGHTMAN AND HARRY C. JONES. Johns Hopkins Univ. Am. Chem. J., 46, 56-112.—In continuation of the work of White and Jones (C. A., 4, 2759), the authors have made similar detns. with a number of other organic acids and present the results in numerous tables and figures. The conclusions reached confirm those of the earlier paper; in addition, the following may be stated: The temp. coeffs. of cond., expressed in cond. units, increase rapidly with diln., and decrease rapidly with rise of temp. for weak org. acids—when not hydrated. When the acids are hydrated, the temp. coeffs. are larger, and their increase with diln. and decrease with rise of temp. both take place at a slower rate. Org. acids with the largest dissociation consts. also have the largest temp. coeffs. expressed in cond. units. J. Johnston.

Surface Effects between Mercury and Certain Solutions and an Electrochemical Method of Estimating Dissolved Oxygen. S. W. J. SMITH AND W. F. WIGGINS. Electrician, 66, 993; J. Soc. Chem. Ind., 30, 371.—The rate of change of e. m. f. resulting from additions of Na<sub>2</sub>S is at first roughly proportional to the amt. of Na<sub>2</sub>S added but suddenly increases rapidly at a critical conc. lying near 0.0004 equiv. Na<sub>2</sub>S per 1. in solns. containing about 0.0011 equiv. O per 1. This effect is temporary and almost disappears within 24 hrs. Thru further addition of Na<sub>2</sub>S a second similar effect appears which is permanent so long as atmos. O is excluded; this is due to the conversion of the Na<sub>2</sub>S into Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> by interaction with the O in the soln. The amt. per 1. of Na<sub>2</sub>S required to produce the effect is thus a measure of the true conc. of dissolved O. A very slight excess is sufficient to alter the e. m. f. by more than 0.5 v.

C. N. MOORE.

A New Determination of the Electromotive Force of the Weston Normal Cell. H. HAGA AND J. BOEREMA. Groningen. Proc. Konink. Akad. Amsterdam, 19, 587 (1910); Elektrotech. Anz., 28, 315.—The materials used in the construction of the cells were prepared as follows: The Hg was distilled first at reduced pressure with air bubbling thru it and then several times in vacuo. The amalgam containing 12.5% Cd (by wt.) was prepared electrolytically using Cd from Kahlbaum as anode. CdSO. (Kahlbaum) was recryst. a few times before using. The Hg<sub>2</sub>SO<sub>4</sub> was prepared from an acid soln. of HgNO, formed from conc. HNO, and Hg. The HgNO, was poured in a finely divided stream into hot dil. H2SO4, while shaking vigorously. The ppt. was filtered, washed twice with dil. H, SO, and then several times with a neutral soln. of CdSO<sub>4</sub>. The e. m. f. of the cell was compared with the drop in potential across a known resistance caused by a known current. The value of the resistance in international ohms was found by comparison with 2 one-ohm standards tested at the Reichsanstalt before and after the investigation. The current was measured by means of 2 tangent galvanometers; the major part of the paper is devoted to the discussion of this part of the work. In all 31 cells were used and the mean value found for their e. m. f. at 17° was 1.0183s v. (international ohm × c. g. s. amp.). FRANK WENNER.

A New Determination of the Electromotive Force of the Weston Normal Cell.

R. DE BAILLEHACHE. Arch. sci. phys. nat., 31, 185-208.—A full account of the above investigation of Haga and Boerema.

J. JOHNSTON.

Capillary Rise of Amines, Phenols and Aromatic Hydroxy Acids. Z. H. SERAUP AND E. PHILIPPI. II Chem. Lab., Univ. Vienna. *Monalsh.*, 32, 353-72; cf. C. A., 4, 1925; 5, 17, 2021.—With NH, the height reached decreases as the conc. decreases.

For HONH, and N,H, solns. it is much greater than for the strong alkali hydroxides. In the case of the aliphatic monamines and the corresponding quaternary bases, the nature of the alkyl is without influence. The effect of the number of substituting alkyl groups may be expressed by saying that the primary and secondary monoamines give substantially similar results, which are decidedly higher than those afforded by the alkali hydroxides; the quaternary bases show smaller rises, while the tertiary monoamines resemble NH, and give values which differ according to whether the detn. is made in an open or closed tube. The diamines, for equiv. conc., rise less than the monoamines. Regularities are less marked in the case of the aromatic amines, but, in general, the monoamines rise higher than the diamines. In the case of the toluidines the values decrease in the order o-, p- and m-. The values for o- and m-phenylenediamine are nearly identical and are much higher than those for the p-compd. The hydrochlorides give values similar to those obtained for the free amines if the color reaction for the amine is employed as indicator, but the acid reaction (with azolitmin) takes place much lower; it is, however, considerably higher than that given by pure HCl. In the case of the phenols, the influence of conc. is less marked. For PhOH, pyrocatechol and hydroquinol the rises are practically equal; resorcinol, pyrogallol and phloroglucinol give lower values, decreasing in the order named. The results for the aromatic hydroxy acids differ completely, according to whether azolitmin paper, or white paper with a suitable reagent is employed. The zone of color is lower in the former case. The white paper (containing a reagent) gives a well marked color at about the height of that shown by the azolitmin paper, and a feebler color at a higher point. This is probably caused by the ash of the paper neutralizing some of the acid: the resulting salts then increase the height which the soln. attains. Paper practically free from ash gave results which were essentially the same as those observed with azolitmin paper. Na salicyla e gives the same rise as that shown by the acid with paper containing ash. The rise of all the acids employed was very similar and decreases rapidly with the conc. J. BISHOP TINGLE.

Heats of Reaction in Non-aqueous Solutions. J. HOWARD MATHEWS. Phys. Chem. Lab., Univ. Wisconsin. J. Am. Chem. Soc., 33, 1291-1309.—Measurements of the heat of reaction of 1 mol. pyridine with 1 mol. HOAc were made (a) in the absence of a solvent (b) with one or both the substances in soln. in H<sub>2</sub>O, Me<sub>2</sub>CO, EtOAc, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>2</sub> or CCl<sub>4</sub>; further the heat of soln. of the resulting acetate in each of the above solvents was detd. The considerable differences in the values found cannot be explained as being due either to electrolytic dissociation of the acetate, or to differences in the heat of soln. There is no apparent relation between the chem. nature of the solvent and the heat effects observed.

J. JOHNSTON.

The Vapor Pressure of Hydrates, Determined from their Equilibria with Aqueous Alcohol. H. W. FOOTE AND S. R. SCHOLES. Sheffield Chem. Lab., Yale Univ. J. Am. Chem. Soc., 33, 1309-26.—The authors have detd. the conc. of aq. alc. in equil. at 25° with each of a number of pairs of hydrates, resp. hydrated and anhydrous salt. The corresponding equil. pressure is then the partial H<sub>2</sub>O vapor pressure of the particular conc. of aq. alc. This was read from a curve derived from measurements of the total vapor pressure of a series of H<sub>2</sub>O-EtOH mixtures at 25°, using an aspiration method.

J. JOHNSTON.

Dissociation of Cupric Bromide and some Forms of Glass Manometer. C. G. Jackson. Univ. Breslau. J. Chem. Soc., 99, 1066-71; Proc., 27, 45.—There are described 3 forms of glass manometer, such that the vapor whose pressure is to be measured comes in contact with glass only. With the manometer—which differs only in detail from that described by Johnson (C. A., 2, 1085)—measurements of the dissociation pressure of CuBr<sub>2</sub> at a series of temps. were made: for example, at 166°, 3.1 mm.;

at 281°, 614.5 mm. Pressures calc. by the formula  $ln(p_1/p_2) = -Q(T_1 - T_2)/RT_1T_3$  agree well with those found by expt. The graph of  $T \log p$  as a function of T is approx. linear. The mean value of Q calc. from the above eqn. is 22400 cal. D. M. LICHTY.

Separation on the Appearance of a Solid Phase. A. Smits. Amsterdam. van Bemmelen Gedenkboek, 434-40; cf. C. A., 5, 1863.—A theoretical paper on the equil. of two liquids when a solid phase appears.

J. JOHNSTON.

Liquidus Surface of the Ternary System Composed of the Nitrates of Potassium, Sodium and Calcium. A. W. C. Menzies and N. N. Dutt. Kent Lab., Univ. Chicago. J. Am. Chem. Soc., 33, 1366-75.—The temp. of disappearance (and of appearance) of crystals on heating (or cooling) was detd. for about 70 mixts. of the pure salts with an accuracy of  $\pm 1^{\circ}$ . The numbers given below represent the temps. (based on the Pt thermometer calibrated at 0°, 100° and S b. p. (assumed to be 445°)) corresponding to the mixts. indicated (the conc. of which is expressed in mol. percentages): pure NaNO<sub>2</sub>, 315.1°; pure KNO<sub>3</sub>, 346.3°; [20 NaNO<sub>4</sub>, 45 KNO<sub>4</sub>, 17.5 Ca(NO<sub>4</sub>)<sub>2</sub>], 175°. Binary eutectics: [50 NaNO<sub>3</sub>, 50 KNO<sub>4</sub>], 230°; [66.6 NaNO<sub>3</sub>, 33.4 Ca(NO<sub>4</sub>)<sub>2</sub>], 236.5°; between KNO<sub>4</sub> and Ca(NO<sub>3</sub>)<sub>2</sub> it is below 220° at approx. 60% KNO<sub>3</sub>. J. Johnston.

Catalysis. J. BÖESEKEN. Delft. van Bemmelen Gedenkboek, 386-91; cf. C. A., 4, 1484.—The author maintains his view that catalysis can not be explained by the assumption of intermediate reaction products, and discusses the relation between the amt. of catalyst employed and the velocity of the reaction induced, emphasizing the facts that the catalyst is always itself altered to some extent and that a certain small amt. of catalyst is always necessary before the reaction will go on. J. JOHNSTON.

Catalytic Phenomena. J. BÖESEKEN. Chem. Weekblad., 8, 555-79.—A lecture, in which a brief account of our present knowledge of catalysis is given. J. J.

Absorption in a Solution of Several Substances. H. FREUNDLICH AND M. MASIUS. Physik. Chem. Inst., Leipzig. van Bemmelen Gedenboek, 88-101.—The absorption by C was studied in mixed solns. of oxalic acid with AcOH, succinic, and benzoic acid, and in aq. solns. of aniline salts. The following regularities were observed. The absorption equil. is rapidly attained from either side. Both substances are absorbed, that which is absorbed in greater amt. from its pure soln. being also preponderantly absorbed from the mixed soln. Each substance diminishes the absorption of the other; the mutual influence is such that the diminution in the amt. of absorption of one substance increases with the extent to which the second substance is absorbed. The absorption isotherm holds fairly well for a given substance, so long as its absorption is not influenced to any large extent by addition of a second substance. The absorption by C of the aniline salts from aq. soln. (in which they are hydrolyzed to a considerable extent) is such that the base and acid are not absorbed in equiv. amts., the exact amt. of each absorbed depending on the particular acid associated with the aniline. In the absorption of a salt it is well known that there is in many cases an apparent separation of anion and cation, so that the soln. of a neutral salt is no longer neutral after absorption has taken place; this is attributed to the (assumed) presence in the C of a previously absorbed electrolyte or ion, which by reacting with the substance under investigation produces the phenomena observed. J. JOHNSTON.

The Absorption of Methylene Blue and Crystal Ponceau by Carbon in its Dependence on the Temperature. L. Pelet-Jolivet and H. Siegrist. Lausanne. von Bemmelen Gedenkboek, 158-62.—Freundlich found that the adsorption of AcOH by C was decreased by rise of temp. (C. A., 1, 1094), while the author and Grand (C. A., 1, 3058) found that the absorption of methylene blue (a basic dye) by C and by SiO<sub>2</sub> was increased by rise of temp. The effect of temp. (up to 70°) upon the absorption of crystal ponceau (an acid dye) is now studied, with the result that its behavior is

similar to that of methylene blue. This shows that the temp. coeff. of absorption of dyestuffs by C is independent of the acidity of the dyestuff.

J. JOHNSTON.

Colloids and Permanent Modifications of Substances. PIERRE DUHEM. Bordeaux. von Bemmelen Gedenkboek, 1-6.—Thermodynamical principles cannot be applied directly to colloidal systems, because each change in the system is in general accompanied by a permanent change in the colloidal material. In illustration of this point, the author cites his own work on the emission and absorption of H<sub>2</sub>O vapor by colloid matter (J. Physic. Chem., 4, 65 (1900)) and that of Manville (C. A., 2, 33), who found that the conditions and products of oxidation of amorphous C depend upon the previous history of the C.

J. JOHNSTON.

Dialyzability of Dyestuffs. W. BILTZ AND F. PFENNING. Clausthal i/H. von Bemmelen Gedenkboek, 108-20.—A tabulation and discussion of the results of dialysis expts. with a large number of dyes; the conclusions have been published elsewhere (C. A., 5, 2998).

J. JOHNSTON.

Colloids. NICOLA PAPPADA. Cremona. van Benmelen Gedenkboek, 288-96.—
A brief account of current views on the colloidal state.

J. J.

The Viscosity and Fluidity of Emulsions, Crystallin Liquids and Colloidal Solutions. EUGENE C. BINGHAM AND GEORGE F. WHITE. Richmond Coll., Richmond, Va. J. Am. Chem. Soc., 33, 1257-75; cf. C. A., 4, 1565; 5, 1699.—From detns. of the viscosity of phenol-H<sub>2</sub>O mixts. in the neighborhood of the crit. soln. temp., and from a discussion of the existing data for analogous systems it is concluded that when liquids form a thoroly homogeneous mixt. their fluidities will be additive; but that the fluidity of the mixt. will be less than would be the case in an homogeneous soln., whenever the mixt. is not homogeneous, as by the formation of an emulsion, a colloidal soln., or a cryst. liquid.

J. JOHNSTON.

Viscosity of Colloidal Solutions. R. O. Herzog. Z. Chem. Ind. Kolloide, 8, 210-1.—According to Einstein, "when tiny rigid spheres are suspended in a liquid, the coeff. of viscosity of that fluid is increased by a fraction which is equal to the total vol. of the spheres suspended in unit vol. of liquid, provided this total vol. is very small." The size of the particles or degree of dispersion of a colloidal soln. does not, therefore, directly det. its viscosity. The viscosity is detd. by the quantity of dispersion medium bound by the particle and fixed by the properties of the two disperse phases. The reduction in viscosity of suspension colloids on standing is not directly due to a change in the size of the particles but to the reduction in the amt. of the dispersion medium held by the particles. This can, however, be brought about indirectly (I) by a change in the size of the particles, the particles on aging running together and reducing the active surface; (II) by the expression of the dispersion medium from the gradually diminishing particle; (III) by a qual. change in the surface of the particle (by adsorption of electrolytes or such like).

D. Spence.

Viscosity of Colloidal and Non-colloidal Liquids. Ludwig Dienes. Univ. Budapest. Biochem.  $Z_{\circ}$ , 33, 222-4.—Expts. were made with  $H_2O$ , solns. of NaCl, urea, cane sugar, egg white, Witte peptone, and Me violet, using von Liebermann's modification of Coulomb's app.  $(C.\ A.,\ 5,\ 2992)$ . In the case of non-colloids it was found that the log. decrement of the amplitude of vibration increases with the depth the disk is sunk in the liquids, while with colloidal solns. this value is greatest near the surface.

F. J. Birchard.

Osmotic Pressure of Colloids. JACQUES DUCLAUX AND E. WOLLMAN. Compt. rend., 152, 1580-3.—The authors measured the osmotic pressure of solns. of nitrocellulose in acetone in concs. up to 20%, using a membrane of denitrified collodion. The ratio of pressure observed to conc. increased rapidly with the conc. This diverg-

ence from constancy cannot be accounted for by an equation analogous to that of van der Waals; it is, however, in agreement with the theory put forward by Duclaux (C. A., 4, 405).

J. JOHNSTON.

Theory of Swelling. K. SPIRO. Univ. Strassburg. van Bemmelen Gedenkboek, 261-6.—It is shown that 2 of the factors which influence the swelling or contraction of a jelly (e. g., of agar or gelatin) are: (1) the original shape of the piece of jelly, which indicates the action of forces at the surface of the jelly; (2) the initial conc. of the jelly. Thus 2 jellies of similar shape, but of different agar content, behaved differently towards aq. solns. of a number of substances. Isosmotic solns. of NaCl and CaCl<sub>2</sub> give rise to swelling or contraction effects of different magnitudes. The importance of the phenomena in connection with changes in living cells is pointed out. J. Johnston.

Phenomena Observed in the Drying of Gels. RAPHAEL E. LIESEGANG. Frankfurt a/M. van Bemmelen Gedenkboek, 33-5.—The author endeavored to prepare dry gelatin films containing a sol. salt uniformly distributed thruout the film, but found it impossible to do so, even with salt solns. so dil. that no crystn. took place. The lack of uniform distribution is due to a diffusion of the salt from the edge to the middle, from dry to moist places (cf. C. A., 5, 404, 1703).

J. JOHNSTON.

"Pyrosols": Colloidal Phenomena in Glowing Liquids. RICHARD LORENZ. Frankfurt a/M. van Bemmelen Gedenkboek, 395-8.—A brief account of the observations on metallic fogs in fused salts (cf. C. A., 5, 2456) and the allied phenomena of the colors produced in glass by Au, etc., for which the author proposes the term "pyrosol."

E. J. WITZEMANN.

The Relation of Artificial Phosphorescent Stone and Colloidal Solutions. Ludwig Vanino. van Bemmelen Gedenkboek, 392-4.—Phosphorescent masses are analogous in many respects to colloidal solns. and might well be classed as colloidal masses rather than as solid solns.

E. J. Crane.

Mode of Conduction in Gases Illustrated by the Behavior of Electric Vacuum Valves. OLIVER LODGE. Phil. Mag., 21, 1-8.—A series of expts. extending over a number of years yield the following generalizations: (1) The current in vacuum tubes is mostly conveyed unobtrusively by positively charged carriers or atoms which travel from anode to cathode as best they can, readily taking a roundabout path if necessary, i. e., if any serious obstruction exists in a more direct path. (2) From the surface of the cathode a bombardment of negative corpuscles or electrons occurs from every place at adequate tension, and issues everywhere perpendicular to the surface without regard to destination. At high vacuums these projectils travel with prodigious speed for a considerable distance, and they have the effect of blocking the path by driving back any small bodies, such as atoms, advancing in an opposit direction. The negatively charged projectils carry only an insignificant proportion of the whole current, tho they may assist in propelling uncharged atoms towards the anode, where they can become positive ions. (3) Circulation of material in the tube is necessary, so as to maintain a constant supply of positively charged ions and to enable them to act as carriers. R. F. EARHART.

Origin of Spectra. Frank Horton. Phil. Mag., 21, 214-9.—The particular spectrum discussed is that produced by Hg, which exhibits 5 distinct line spectra. The author outlines the views of Stark on the origin of the spectra and Wien's criticism of Stark's theory. Evidence is adduced in support of the theory that ionized Hg vapor absorbs the rays it emits, whereas vapor in the normal condition does not. J. J. Thomson, in his expts. on positive rays, has shown that Hg vapor may have elec. at. wts. of 800, 200, 100, and 66. The author believes that the vibrations of the heavier systems are the origin of band spectra while the systems of smaller mass pro-

duce line spectra. The many-lined spectrum is produced when a large amt. of energy is transmitted thru the vapor.

ROBT. F. EARHART.

Spectroscopic Proof of the Repulsion by the Sun of Gaseous Molecules in the Tail of Halley's Comet. Percival, Lowell. Proc. Am. Phil. Soc., 50, 254-60.—A great many photographs and spectrograms of Halley's comet were made at Flagstaff, Ariz., during 1910. Photographs of the tail showed knots or condensations, which were moving away from the head. Measurement proved that the motion was accelerated; indicating a repulsive force, presumably that of light-pressure, away from the sun. Spectrograms show that by far the larger % of the light from the tail is emitted by the matter constituting the tail, and that this light was due to CO and CH4. Light reflected from solid particles would show a continuous spectrum; and altho there was a certain amt. of continuous spectrum present, its total light was only a small fraction of the whole. The accelerated motion of the knots in the tail is hence taken as a proof that light-pressure acts on gaseous mols.

J. A. Anderson.

Increase of Electric Conductivity of Liquid Dielectrics on Illumination with Ultraviolet Light. G. Szivessy and K. Schäfer. Stuttgart. Ann. Physik, 35, 511-23.

—The increased cond. of liquid dielectrics caused by the action of X-rays and by the 7-rays of Ra has been studied by J. J. Thomson, Curie, and Jaffé. The author investigates the effect of short ultraviolet rays on very pure paraffin oil. The light was furnished by a quartz Hg lamp and was admitted thru a thin quartz plate to the oil, which was placed between the plates of a condenser (guard ring type), one plate of which was charged, while the other was connected up to an electrometer. The current thru the oil was measured before and after illumination, the difference giving the ionization current. This current was found to increase with the potential applied, but in such a manner as to indicate the existence of a satn. current, as is the case with the ionization current in gases. The whole effect is due to the shortest light waves transmitted by quartz; for it was found that by placing a plate of mica only 0.2 mm. thick in the beam of light, no effect was observed.

J. A. Anderson.

Second Spectrum of Hydrogen in the Extreme Red. F. CROZE. Compt. rend., 152, 1574-6.—The author has succeeded in photographing the second H spectrum from 6582 to 8027. A table of wave lengths and intensities is given. No marked regularities in the distribution of lines were observed. Discussion of the results is reserved for later communication.

C. M. SPARROW.

Measurement of the Intensities of the Various Components of a Complex Radiation. H. Buisson and C. Fabry. Compt. rend., 152, 1838-41.—A method of obtaining the energy curve of a complex radiation by measuring (a) the total energy, (b) the total energy transmitted by various absorbing screens. From a knowledge of the absorption coeff. of the screens for various wave lengths the intensities of the various components can be calc. As an illustration an account of some measurements on a quartz Hg lamp are given.

C. M. Sparrow.

Spectrum of the After-glow (Spectroscopic Investigations in Connection with the Active Modification of Nitrogen, I). A. Fowler and R. J. Strutt. Imp. Coll. Sci. and Tech., S. Kensington. Proc. Roy. Soc. London (A), 85, 377-88; cf. Strutt, C. A., 5, 2601.—In the present work the authors have photographed the spectrum from wave length 2200-7000 Å. units. The spectrum shows groups of bands which may be divided into 4 classes. (1) Consists of bands in the red, yellow and green, which are identical in position and structure with portions of the first positive group of N; they thus consist of a special enhancement of these portions. (2) Bands between 4312 and 2503, corresponding to faint bands in the uncondensed discharge of N. (3) has already been shown by Lewis to be identical with the third positive N group. (4)

consists of seven complex bands, lying between 2904 and 2256, which are new and which appear to be characteristic of the after-glow. Measurements on the bands and formulas showing the series relationships between the members are given. C. M. S.

The Valence Hypothesis Applied to Fluorescence. J. STARK. Aachen. Z. Elektrochem., 17, 514-7.—Diagrams are given of the author's conception of atoms of various valencies, showing the positive charges on the surface of the atom and the bonding between these and the valence electrons, i. e., those negative electrons which define the valency of the atom. In the electronegative atom part of the positive charges are not satd., i. e., not bonded to the valence electron or electrons. In the case of an atom such as Ne, which does not enter into combination with other atoms, the negative electrons are very firmly held and there are no free bonds. No atom ever loses positive charges ( $\alpha$ -rays), except when the at. breaks up completely, as in the case of radioactive elements. Whenever a valence electron absorbs light-energy the latter is transformed partly into kinetic and partly into potential energy and the bond between negative electron and surface of the atom is weakened or entirely destroyed. As soon as the light source is removed and the atoms are no longer under strain, the dissociated, or partly dissociated, electrons are attracted back to their original positions in the atoms and the stored potential energy is converted partly into kinetic energy and partly back into light energy (fluorescence). The dampening effect of foreign gases such as H, CO, Cl, etc., on the intensity of the fluorescence is due to the smaller % of potential energy transformed back to light energy, owing to combinations between the dissociated valence electrons of the fluorescent atoms and the atoms or mols. of the added gas, such as Cl. This hypothesis has been substantiated by expts. of Elston, Franck, Wood, Ley, and others. C. G. FINK.

Stokes' Law and a General Relation between Absorption and Phosphorescence. L. Brüninghaus. Compt. rend., 152, 1578-80.—The original form o. Stokes' law, which stated that the wave length of the fluorescent light was always greater than that of the exciting light, has been shown to be inaccurate. A modification by Lommel, applying to substances having a single band of fluorescence, has been found to be generally valid for such substances. This law states that the max. of the fluorescence band is always of greater wave length than the max. of the absorption band. The author now extends this statement to cover the case of multiple bands, and announces as generally valid for such cases the law that the phosphorescence maxima are always of greater wave length than the adjacent absorption maxima; that is, to each system of absorption bands corresponds a system of phosphorescence bands which are shifted relatively toward the red end of the spectrum. A number of typical examples are given.

C. M. Sparrow.

Magnetization of Ferromagnetic Substances above the Curie Point. PIERRE WEISS AND G. FOEX. Arch. sci. phys. nat., 31, 5-19, 89-117; J. physique, [5] 1, 274-88.—According to Weiss' theory of the mol. magnetic field, the equation  $\chi(T-\theta)=C$  should hold, where  $\chi$  is the sp. susceptibility,  $(T-\theta)$  the excess of temp. above the point at which spontaneous ferromagnetism disappears (the Curie point), and C a const. The validity of this equation was tested by expts. over a considerable temp. range on Fe, Co, Ni, (artificial) magnetite, pyrrhotite, and a series of Fe-Ni alloys. The results are exhibited in curves, in which T is plotted against  $1/\chi$ . The curve for magnetite consists of 4 straight lines, indicating the presence of 4 different forms. For Ni  $(\theta=372^\circ)$  it is a straight line up to 1100°; for Co  $(\theta=1150^\circ)$  it is also a single straight line. In Fe the discontinuities at 920° and 1395° separate the  $\beta$ ,  $\gamma$  and  $\delta$  regions. For pyrrhotite  $\theta$  appears to be 319°; but in this case  $\chi$  is nearly independent of the temp. From the results on the Fe-Ni alloys it is concluded that at high temps. they form two series of solid solns., extending on the one hand from Fe to the comp.

Fe<sub>2</sub>Ni and on the other from Fe<sub>2</sub>Ni to Ni; and that in a solid soln. of 2 magnetic metals the Curie constant is additive, tho it is not in a chem. compd. The above results indicate the existence of the compd. Fe<sub>2</sub>Ni.

J. JOHNSTON.

Chromoisomerism (HANTZSCH). 10.

Organic Ammonium Halides (HANTZSCH, HOFMANN). 10.

Viscosity and Diastatic Reactions (ACHALME). 11.

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#### 3. RADIOACTIVITY.

#### HERMAN SCHLUNDT.

Atmospheric Electricity over the Ocean. G. C. SIMPSON AND C. S. WRIGHT. Proc. Roy. Soc. London, (A) 85, 175-99.—The observations were made during a voyage from England to New Zealand. The plan of work embraced investigations of (1) the elec. potential existing over the ocean; (2) the quantity of radioactive products in the air; (3) the number of free ions over the ocean; (4) the ionization of the air in a closed vessel, with the object of detg. the presence or absence of a penetrating radiation over the sea. During the whole voyage, the potential gradient was found to be, without exception, positive on fine days. During one period of steady rain it was negative. The curves showing the variation of potential gradient indicate that a daily max. exists in the evening, and a minimum soon after mid-day, over the South Atlantic and South Indian oceans within 40° of the equator. The radioactivity expts. show that the quantity o Em. over the sea is less than over the land, and that the amt. of active matter is more const. The free ions in the air increased from 10° to 40° S. latitude, and the ratio of the + to -- ions decreased from 1.36 to 1.10; these results are related to the prevailing winds in different latitudes. The natural ionization in a closed Zn vessel of 27 l. capacity showed no regular daily variation. A slight dependence of the natural ionization upon barometric pressure was observed, a high barometer giving a low value for q, the number of ions per cc. of gas. The lowest value of q per sec. was 4. A part of the normal ionization measured was found to be due to radioactive products

deposited on the ship. The penetrating radiation from the soil at Matjesfontein produced 6 ions per sec. per cc. in the Zn cylinder used aboard the ship. H. SCHLUNDT.

Transformation of Actinium Emanation. H. GEIGER. *Phil. Mag.*, 21, 201-4. —The character of the radioactive products was detd. by the range method, using the scintillation test as a detection. The Act. Em. is shown to consist of 2 products, each of which emits  $\alpha$  rays. The Em. has a 4-sec. period and emits  $\alpha$  rays whose range is 5.7 cm. The second product emits an  $\alpha$  ray with a range of 6.5 cm. and has a period of  $1_{600}$  sec. The latter product carries a positive charge and is probably a solid.

The Behavior of Radium Emanation at Low Temperatures. R. W. BOYLE. Mag., 21, 722-33.—Below —127° the vapor phase of Ra Em. has been traced only by a flow method devised by Rutherford, in which the amt. of Em. was so small that a layer of the condensation over any considerable area would be less than mol. thickness. B. employs a direct method involving much larger quantities of Em. The Em. was condensed in the tip of a capillary tube, the upper part of which was bent twice at right angles and closed at the upper end with a thin mica plate above which an ionization chamber was mounted. This was sealed off at a charcoal vacuum. At the temp. of liquid air the ionization in the chamber became small after 4 hrs., during which time the active deposit in the upper part of the tube decayed. With a rise in temp. some of the Em. would vaporize and an active deposit be formed in the upper part of the tube, thus producing an ionization in the chamber. The double bend in the tube was for the purpose of cutting off particles projected from the condensed Em. The expt. shows that the vapor phase can be traced as low as -180°, for which temp. the state of the condensed Em. was not known. Expts. to det. the vapor pressure between -180° and -127° were not successful. ROBT. F. EARHART.

A Method of Investigating the Quantity of Radium in Rocks, Minerals, etc. J. Joly. Phil. Mag., 21, 134-50.—The usual method of analysis, that of soln. involves a long and tedious process. Frequently, reactions may occur which render the subsequent detn. of the Em. by ebullition impossible. A method of measuring the Em. is proposed and tested wherein powdered rock samples are fused with Na<sub>2</sub>CO<sub>2</sub> and K<sub>2</sub>CO<sub>3</sub> in a specially constructed Pt furnace (2 forms are described). The gases evolved are collected in a gas bag and later tested with an electroscope. The Em. product ranges higher (sometimes 46%) than in the soln. methods.

R. F. EARHART.

Radioactivity of some Igneous Rocks from Antarctic Regions. ARNOLD L. FLETCHER. Phil. Mag., 21, 770-3.—Samples taken from the same locality and analyzed by the soln. method showed remarkable agreement for both Ra and Th content, indicating their origin from a common magma. The rocks from the Mt. Erebus region showed this characteristic tho differing chemically and petrographically. The results do not confirm the conclusion of Farr and Florence that the radioactivity depends roughly upon the acid or basic character of the rock. A sample of red Kenyt, colored probably by infiltering geyser action, showed an unusually high radioactivity. Both Ra and Th content were high.

Note on the Paper of E. Rutherford and H. Geiger on the Probability Variations in Distribution of Alpha Particles. *Phil. Mag.*, 21, 198-200.—The theory of ideal frequency curves devised by Karl Pearson is applied to the problem presented by Rutherford and Geiger (C. A., 5, 1231). Comparison between theory and expt. is indicated for  $\frac{1}{6}$ -min. intervals. The agreement is better for the  $\frac{1}{6}$ -min. interval. R. F. E.

Brit., 778, Jan. 11, 1911. C. SCHMIDT, Alum Works, Greienwalde a/Oder, Ger. Mfg. a radioactive material by mixing a radioactive substance with a binding agent and a combustible substance and then subjecting the mixt. to heat.

#### 4. ELECTROCHEMISTRY.

C. G. FINK.

The Hydroelectrica Espanola. M. NEUSTAETTER. Elektrotechn. Z., 32, 535, 561.—An illustrated description of the large plant on the Jucar River.

L. K. MULLER.

The Electric Furnace, its Place in Siderurgy. P. McN. Bennie. Proc. Eng. Soc. Western Penn., 26, 487.—Electric-furnace production of pig iron and the new plant of the Noble Elec. Steel Co., Shasta Co., Calif., are described, as well as elec. steel production and the various furnaces used. Cuts of the most important furnace types are given, together with tables of analyses and physical tests of the products (cf. C. A., 5, 629).

HORACE C. PORTER.

Electric Iron Ore Smelting at Trollhaettan. I. J. A. LEFFLER AND E. ODELBERG. Engineering, 91, 778, 811, 846; Met. Chem. Eng., 9, 368-71, 459-63; Eng. Mag., 41, 846.—A very full description of the construction and working of this important plant. In the furnace, charcoal is used for reduction. Four electrodes project thru the crucible roof into the widened crucible or hearth at the bottom of the shaft. Gas is drawn from the downcomer and forced into the crucible to cool the roof and aid in heating and reducing the ore as it passes up the shaft. The information so far obtained shows the furnace to be most suitable for steel making. The tests on the manuf. of pig Fe are not yet completed.

A. S. EASTMAN.

The Reducing Fusion of Oxide Ores in the Electric Furnace. W. BORCHERS. Metallurgie, 8, 246-8.—The chief difficulty encountered in the reduction of Ti-Fe ores in an elec. furnace is the fact that the fused oxide is sepd. more or less from the specifically lighter reducing agent by an intervening layer of slag. If an Fe-Ti ore is melted without slagging fluxes with an insufficient amt. of reducing agent, a part of the FeO is kept back as slagging agent for the TiO<sub>2</sub>; by choosing the right amt. of C a very difficulty fusible acid slag rich in TiO<sub>2</sub> is produced. This acid titanate when reduced with the idea of forming an Fe-Ti alloy yielded a mixt. of iron titanide, Ti carbide, free Ti or even free C according to amt. of reducing agent employed. This mixt. of titanides and carbides proved to have strongly reducing properties and is so heavy that it does not, like C, tend to float on top of the charge. In other words, the prep. of this reduction product has served to weight the reducing C and to make it so reactive that the reduction of difficult oxide ores takes place much more quickly and much more cheaply. W. T. HALL.

The Electric Furnace for the Manufacture of Iron and Steel. JAMES LYMAN. Chem. Eng., 13, 250.—The advantages of the elec. furnace are discussed, and the Frick elec. reduction arc furnace, the Héroult furnace, the Kjellen and Röchling-Rodenhauser furnaces are described and illustrated.

A. S. EASTMAN.

Production of Mild Steel in the Electric Fu nace. F. Doubs. Stahl u. Eisen, 31, 589-92; thru J. Soc. Chem. Ind., 30, 545.—After investigation of the workings of the Héroult, the Nathusius and the Kellar furnaces, of which tables of cost, and of comp. of charges and product are given, the author concludes that with cheap current the elec. furnace can compete with small open-hearth furnaces even with cold charges. For production of crucible tool steel and even of cupola Fe, elec. melting is not more expensive where coke is high in price. Round electrodes are advocated and, especially with large furnaces, electrodes and gear should be supported independently of the furnace, which should be arranged for tilting through an angle of 90°. The durability of hearths with imbedded electrodes is stated to be excellent. L. W. Spring.

Electric Crucible Furnace for Melting and Pouring Metals. W. BORCHERS. Metal-

lurgie, 8, 209-11.—G is the melting and pouring vessel and consists of a sheet-iron mantle lined with a heat-resisting material such as burnt clay.



mantle lined with a heat-resisting material such as burnt clay. quartz, burnt magnesia or dolomite, etc. The bottom electrode, B, is made of the same metal as that it is desired to melt in the crucible. Its shape is that of a truncated cone with the lower end hollowed out somewhat to permit the introduction of the cooling body, C. The latter is clamped in a holder, H, which can be raised or lowered and serves, at the same time for making connection with the elec. current. The other electrode, E, is usually made of C and is introduced through the cover D, which is composed of one of the above-mentioned heat-resisting materials and rests in a suspended iron frame that can be raised or lowered. To prevent losses from leakage and consequent damage to the piping, an Fe plate, T, may be placed over the cooling body and should be coated with a thin layer of the same material as that with which the crucible is lined. W. T. HALL.

Electric Furnaces for Molten Materials. C. HERING. Met. Chem. Eng., 9, 371-3; J. Frank. Inst., 172, 55-72; cf. C. A., 5, 2032.—The following ideal features of a furnace are discussed: economy in power; rapidity of treatment; heating from the bottom instead of from the top; no higher temp. than that actually required; rapid and uniform distribution of the heat; rapid circulation to produce greatest possible homogeneity of product; largest possible surface of contact between the metal and the slag when the treatment is dependent upon the chem. action of the slag; heat transmitted from the metal to the slag rather than the reverse; greatest possible reduction of the losses due to the electrodes; possibility of adding fuel heat; ease of control; reliability of operation; simplicity and cheapness of construction and operation. A. S. EASTMAN.

The Nitrate Industry at Notodden. MAIZIÈRES. Engrais, 25, 1048-9.—A review of the electrochemical processes for producing nitrates and a report of the output of the plants at Notodden.

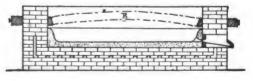
E. C. LATHROP.

Aluminium Nitrogen. Anon. Monotsh. Landw., 3, 264; thru Biedermann's Zentr., 39, 640.—Serpek's method is briefly described. A current of air deprived of part of its O by passing over glowing coke is conducted through an oven containing a mixt. of clay and charcoal heated by elec. The aluminium N thus obtained is produced with an expenditure of only 1/2 of the elec. energy required for the production of a like amt. of lime N.

WM. P. GARRETY.

New Electric Resistance Furnace. F. A. J. FITZGERALD. Trans. Am. Electrochem. Soc., 19, 273-84.—An elec. furnace, 5.5 by 14.5 ft. by 4 ft. high, constructed primarily for Zn reductions according to the Imbert method: ZnS + Fe = Zn + FeS. This reaction takes place fairly completely above 1000°, especially when a fluid bath, one part  $Fe_2O_3$  and 3 parts  $FeS_2$ , is used. In actual operation the bath had a temp.

of 1500°. The resister (R in the figure) is just underneath the roof, connecting with the furnace terminals at either end. The resister has a high contact resistance and is constructed of 71 corrugated C plates of special design. The re-



sistance due to the contacts was found to be 98 per cent. of the total resistance of the resister. Illustrations and photographs are shown. For Zn smelting, the furnace chamber is kept air tight. Two of these furnaces are in practical operation, one at Niagara Falls, the other at Hohenlohehuette, Upper Silesia. H. M. FERNBERGER.

Melting Non-ferrous Metals in an Electric Furnace. C. A. WEERS. Met. Chem. Eng., 9, 363-5.—An elec. furnace for melting Cu, Zn, monel, bronze, and brass. The furnace (no details given) is of the arc type; the heat is transferred to the metal by radiation from the arc. The furnace is entirely enclosed and the atmosphere is neutral. The capacity is about 3 tons of Cu. For Zn smelting, a condensation chamber is attached. Melting 2000 lbs. of Cu scrap required 500 kw-hrs., time 3 hrs. No vaporization of Cu occurred. Castings of monel metal were obtained, practically free from pores. The comparative costs for brass melting are: coke-heated crucible furnace, \$5.65 per ton; direct oil-fired furnace, \$3.86; elec. furnace, \$3.44. Zn metal at the rate of 35 lbs. per hr. was obtained from a dross containing 2% Pb and 6.5% Fe. No loss due to formation of "blue powder"; the product analyzed 99.2% Zn.

L. K. MULLER.

Carbides and Silicides. O. Honigschmid. Z. angew. Chem., 24, 792-3.—Largely a review. Ca silicide is used in fixation of atmospheric N; ferrosilicon is used as a flux for art castings and as a material for evapg. dishes and electrodes.

E. J. WITZEMANN.

Electrolytic Refining as a Step in the Production of Steel. C. F. Burgess. Trans. Am. Electrochem. Soc., 19, 181-4.—The author shows that electrolytic Pe can be produced with a purity as high as 99.97% and with a greater uniformity of product than by other means. With a bath of 40 g. of Fe per 1. as FeSO<sub>4</sub> and 40 g. NH<sub>4</sub>Cl refining operations have been carried on continuously on a small scale for many months, using a current d. of 6-10 amp. per sq. ft. of cathode surface, at a p. d. of 1 volt. The current efficiency is close to 100%. Refining costs are figured at \$20 per ton. Total cost of high grade electrolytic Fe, \$55 per ton.

Polarization in its Relation to the Decomposition Pressure of Electrolytes. O. P. Watts. Trans. Am. Electrochem. Soc., 19, 91-108.—The phenomenon of decomp. voltage is due, not to the resistance of a gas or other film upon either electrode, but chiefly to the counter e. m. f. produced at the electrodes as a result of the impressed e. m. f. This counter e. m. f. is referred to as polarization. The "total" polarization may be resolved into 2 components, the one at the anode, the other at the cathode. The source of the counter e. m. f. is of the same nature as the e. m. f. of any primary or secondary cell.

H. M. Fernberger.

The Outlook in the Chlorine Industry. P. ASKENASY. Chem. Ztg., 35, 609-10.

—The author divides the chlorine factories into 2 classes: (1) Plants where Cl is worked up into chlorate of lime or into liquid Cl. (2) Plants where Cl is used for mfg. other Cl preps., especially for such as are used in the manuf. of coal-tar colors. The industry in Germany is at present in a very dangerous condition on account of an overproduction.

ROBERT KANN.

Operating Costs of Alkali Chloride Electrolysis. V. ENGELHARDT. Chem. Ztg., 35, 573-4, 582-4.—The continual simplification of electrolytic app. for the electrolysis of alkali chlorides has enabled many consumers of Cl to manuf. this product themselves. Apart from the electrolytic chlorate industry the com. chloride electrolysis is divided into 2 general groups: (a) The direct production of hypochlorite. (b) The sep. manuf. of Cl and alkali. The various mfg. processes and operating costs are discussed. On account of the technical advantages (less harmful effect on the fibers, less acid consumption, etc.), the electrolytic bleaching process may be considered as more efficient even if the mfg. costs for the bleaching material are higher. Cl plants in paper and in texcil (especially linen) mills are in general more economic if, instead of the direct hypochlorite manuf., the sep. process of mfg. Cl and alkali, with utilization of the latter, is employed. Under av. working conditions the diaphragm methods are more

advantageous on a large scale than the Hg processes. For new plants the Hg methods can be considered only if a fair price can be obtained for the Cl-free alkali and if very cheap power is obtainable.

ROBERT KANN.

Standard Electrode with Alkaline Electrolyte. F. G. Donnan and A. J. Allmand. Proc. Chem. Soc., 27, 70; J. Chem. Soc., 99, 845.—Combinations of Hg/HgCl<sub>1</sub>-KCl-. NaOH-HgO/Hg were measured at 25 and 0°. Error  $\pm 0.0002$  to  $\pm 0.00011$ . Assuming the Planck liquid potential formula, single potentials are: Hg/HgO—N KOH =  $\pm 0.1100 - 0.00011$  ( $t-25^{\circ}$ ); Hg/HgO—N NaOH =  $\pm 0.1135 - 0.00011$  ( $t-25^{\circ}$ ); Hg/HgO—N/10 NaOH =  $\pm 0.1690 + 0.00007$  ( $t-25^{\circ}$ ). Av. error after several hrs. = resp.  $\pm 0.00019$ , v.,  $\pm 0.00015$ , v.,  $\pm 0.00057$  v. C. W. Hill.

The Cell, Cu/Cu<sub>2</sub>O-alkali-H<sub>2</sub> at 0°. A. J. Allmand. Proc. Chem. Soc., 27, 69; J. Chem. Soc., 99, 840.—The e. m. f. of the cell at 0° is 0.473 v. (0.462 v. calc. by Nernst theorem). The electrode temp. coeff. was found to be:  $O_2/N$  OH' = -0.00076 v. per degree;  $O_2/N$  NaOH = -0.00072 v. per degree;  $O_2/N$  NaOH = +0.0002 v. per degree. Heat of reaction at 18° of  $O_2/N$  OH =  $O_2/N$  Odt. electrochemically, was 27,530, calorimetric value, 27,400.

Dry Cell Testing. W. B. PRITZ. Trans. Am. Electrochem. Soc., 19, 31-46.-The author comments on the non-uniformity of dry cell testing in connection with the adaptability of a cell to a particular service. Two discharge curves, short circuit vs. service capacity, plotted with the same co-ordinates, show that no relationship exists between these tests. A test with actual service conditions proves satisfactory, while the simultaneous discharge of cells in series thru a R, is unfair to the slightly poorer cell. Affecting both of these are conditions of temp., locality, etc. For example: from -10 to 50° the av. short circuit current of one type of cell varied from 13.7 to 22.4 amps. High temps. give longer service for heavy drains than low temps., while the latter are favorable for service with but light drain. On open circuit, cells stored for 10 weeks at various temps. show % drop in amps. varying from 4.4% at 5° to 98% at 75°, the initial amp. being approx. 20. Tests covering 6 months or a year give a safe comparison of different cells, and the rating of most practical advantage to the consumer is one which gives the life of the cell when used for a specific service. Tests for ignition and telephone work as recommended by a Comm. of the Am. Electrochem. Soc. are given. W. L. VAN KRUBEN.

Rapid Charging of Lead Storage Cells Using Chlorated or Perchlorated Sulfuric Acid. G. Schleicher. Techn. Hochsch., Dresden. Z. Elektrochem., 17, 554-69.— A thoro investigation similar to that of Just, Askenasy and Mitrofanoff (see C. A., 5, 2035). The results are in good accordance with the theory of Le Blanc. The addition of perchlorate is carried out to advantage on a factory basis (Lucas, Ger. Pat., 90,446). Preliminary tests were made by the authors on small smooth plates. The gaseous products O and H were carefully analyzed. The formation of Pb(SO<sub>4</sub>)<sub>2</sub> as an intermediate oxidation product was definitly established. The results are very similar to those of Just, et al (loc. cit.). Perchlorate is more efficient than either nitrate or chlorate, since perchlorate is not reduced at the cathode. For com. plates a soln. of 12.2 g. NaClO<sub>4</sub> per l. 4 N H<sub>2</sub>SO<sub>4</sub> is used.

L. K. Muller.

The Tate Bi-functional Accumulator Plate. A. O. TATE. Trans. Am. Electrochem. Soc., 19, 47-52.—Structurally the cell differs from the usual practice in having short, thin Pb and Pb + PbO<sub>2</sub> ribbons 8 in. long, arranged in multiple, in an alternating series of anode and cathode. Current finds entrance at the top and exit at the bottom, thereby equalizing the potential above and below the horizontal centers. The small width of the ribbon, <sup>3</sup>/<sub>8</sub> in., serves to equalize the current d. in a lateral direction. The departure from the heavy grid type is based on the well-

known fact that electrolytic action does not penetrate more than 1.5 mm. and the Tate cell shows only a 0.75 mm. film on the knurled ribbons. The plate is termed "bifunctional," because it contains within itself the positive and negative elements. The equalization of potential, and the extreme shallowness of the active columns, insuring their activity under all conditions of normal operation, in great measure eliminates the cause of sulfating. Due to this construction, cells can be charged in 35 mins. without any resultant physical injury. The capacity on 8 hr. discharge is 40 amp. hrs. and is being improved. The unit weighs 4.5 lbs. with a capacity of 75 watt hrs.

W. L. VAN KEUREN.

An Improved Method of Electroplating. A. ROSENBERG. J. Roy. Soc. Arts, 58, 298-305.—Application of special powder for each kind of plate, with rag or brush, rubbing object in presence of moisture. The powders (trade name, "galvanit") consist of: (1) metal to be deposited, (2) electropositive metal, usually Mg, (3) substance the aq. soln. of which is an electrolyte. The Zn formula is: Zn 15, (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> 5, Mg 1, chalk 10, soapstone 25 parts by wt. Ni requires most care in securing good deposits; formula: Ni-NH<sub>4</sub> sulfate 20, Mg 2 pts. by wt. Zn, Cd, Ni, Co, Sn, Sb, Bi, Au, brass, gun-metal, German silver, Cd-Ag alloy, may be deposited by the same method. The thickness of the deposit is dependent on the no. of applications. The deposit is pure metal. A deposit of Cd on steel requires 30-40 sec.

Modern Tendencies in Electrodeposition. E. Blassett, Jr. Metal Ind., 9, 330.

The Industrialization of Tungsten. I. H Leiser. Chem. Ztg., 35, 665-6.—The author discusses the various W ore localities and methods for producing the metal. Unusual ores are cuproscheelite, stolzite, ferberite, reinite, powellite, raspite and megabasite. Geologically the W deposits are very similar to those of Sn. Hübnerite yields the best concentrate (nearly free from quartz). Ores which are worked up into ferrotungsten must be free from P and S, and to remove the latter, electrolytic sepn. is used. The present total product of W is 6000 tons high conc. ores, but this could be increased to 20,000 tons. The further industrialization of the metal will not offer any difficulties.

The Dielectric Strength of Air. F. W. PEEK, JR. Proc. Am. Inst. Elec. Eng., 30, 1485-1563. C. G. F.

Electrochemical Reduction of Organic Halogen Compounds (BRAND). 10. Electroplating Battery Plates (HUBBARD). 9. Weston Normal Cell (HAGA, BOEREMA). 2.

BILLITER, J.: Die electrochemischen Verfahren der chemischen Gross-industrie. Halle a/S, 28.50 M.

McMillan, W. G.: A Treatise on Electrometallurgy. Lippincott. 459 pp.

NICOLESCU, C.: Beitrage zur Kenntnis der auf elektrochemischem Wege gewonnenen Oxydationsprodukte des Wismuts. Charlottenburg: G. Heydenreich. 8°, I.M.

ZACHARIAS, J.: Elektrochemische Umformer. Wien: A. Hartleben. 262 pp., 5 M.

Brit., 13,946, June 8, 1910. GEN. ELEC. Co., Schenectady, N. Y. An insulating material contg. a condensation product of phenols and aldehydes dissolved in alc. and paraffin oil dissolved in amyl acetate.

Brit., 18,969, Aug. 12, 1910. E. HIGGINS, "Normandie" 36th and Chestnut Sts., Philadelphia, Penna. In the electrolytic reduction of organic bodies, the employment, in the cathode compartment of an electrolytic cell, of a catalytic agent composed of the oxide or hydroxide of Co or Ni, or a mixt. of these compds., and a suitable chem. inert binding material with which said oxide or hydroxide or the mixt, is mixed.

Brit., 20,634, Sept. 5, 1910. SPECIALFABRIK FÜR ALUMINIUM-SPULEN UND-LEITUNGEN, Gross Frankfurterstrasse 137, Berlin, Ger. For securing insulating films, especially oxide and hydroxide films, on metallic coils of Al and its alloys, a material, adequately liquid and capable of hardening to a considerable degree of mechanical resistance, is forced into a previously oxidized coil, this forcing being effected by suitably regulated exterior pressure alone or by the help of a vacuum previously produced in the coil.

Brit., 25,092, Oct. 18, 1910. LANGBEIN-PFANHAUSER-WEREE, Leipzig-Seller-hausen, Ger. Mfg. electrolytic iron deposits by the electrolysis of hot solns. of Fe salts, with or without conducting salts, and with the use of high current densities, by maintaining the electrolyte at a temp. during the electrolysis between 75 and 85° for the purpose of obtaining detachable Fe deposits of any desired thinness and of as large a surface as required.

Brit., 26,045, Nov. 9, 1910. J. Krannichfeldt, 1 Lochnerstrasse, Koln a/R, Ger. Mfg. an electric incandescent body by electrolytically depositing a metal together with some inert material which when heated emits light with some degree of permanence, and drawing off the metal deposit permeated with inert material from the cathode.

Brit., 27,176, Nov. 22, 1910. J. APOZNANSKI, Mjasnitzkaja 27, Moscow, Russia. In the manuf. of highly porous composition plates for electric accumulators, whereby the paste filled plates are dried at a high temp., the paste filled plates, for the purpose of drying them, are arranged between acid-proof cover plates with the object of utilizing the generation of gas which occurs at the raised temp. for producing innumerable pores and simultaneously for hardening the mass.

Brit., 28,834, Dec. 12, 1910. H. P. RASMUSSEN, Dunedin, New Zealand. A composition for electric insulating and other com. purposes, consisting, e. g., of mineral caoutchouc 1, ground mica 28, ground asbestos 50, ground serpentin 50, S 9, shellac 40.

Brit., 29,830, Dec. 22, 1910. A. B. PRILLY, Canton Vaud, Switzerland. Obtaining in an elec. furnace, Bessemer furnace, Martin furnace, melting furnace, crucible furnace, or the like, an ordinary steel or special iron or steel, such as W-steel, Ni-steel, Cr-steel, ferro-nickel, ferro-chrome, etc., by using as raw materials for supplying the said furnace, chem. pure Fe obtained by the electrolytic refining of cast Fe, and chem. pure alloying metals or metalloids, the operation being then reduced to a simple mixing by fusion.

Brit., 5,281, Mar. 2, 1911. ELEKTROCHEMISCHE WERKE, 2-4 Friedrich Karl Ufer, Berlin, N. W., Ger. In carrying out endothermic gas reactions, the flaming arc between 2 or more electrodes disposed opposit 1 another within a cylindrical furnace, is dispersed laterally by a violent gas whirl in such a manner that the light arc is spread out in the form of a closed disc-like flame.

#### 5. PHOTOGRAPHY.

W. B. HOLMES.

Theory and Practice of Intensifying. II. A. SCHULLER. Phot. Rundschau., 25, 163-9.—The intensifiers are grouped into 4 classes: progressive, surface, hyperpro-

gressive and hypoprogressive. The results of a study of Hg, U and Cu intensifiers is given. The formula D = Nd is used where d = the density before and D the density after intensifying and N = a const. which can be calc. Using varying lengths of time the results are plotted graphically. Further work follows. W. B. HOLMES.

Origin of the White on Lumière's Autochrome Plates. A. FORSTER. Z. wiss. Phot., 9, 291-301; Chem. Zentr., 1911, I, 1674.—While the Lumière pigment is not white but a reddish gray, it appears in the finished picture as a pure white. The cause for this must be in the variation of the AgBr film. This is proved by a microscopic study. The white places of starch grains show a fine granulation of dark separated Ag. If these granulations are dissolved by a soln. of KMnO<sub>4</sub> the color again becomes reddish gray. Illuminated with white light, a better white is obtained than previously.

W. B. Holmes.

A Photochemical Method of Investigation. R. E. LIESEGANG. Phot. Chronik, 1911, 314-5.—A plea for a study of new Ag salts for photographic purposes. A suggestion for the preliminary study of new substances is given. The following example serves as a type. The substance is dissolved in a gelatin soln. and a few drops allowed to set on a plate. A drop of AgNO<sub>3</sub> soln. is added and the results observed. The method serves as a qual. test and indicates whether a further study is desirable. W. B. H.

The Relationship between the Size of the Particle and the Color of the Image. C. Jones. Phot. J., 51, 159-74; J. Soc. Chem. Ind., 30, 712.—Particles of Ag, suspended in a gelatin film, were enlarged by successive treatments with HgCl, and Fe oxalate until they were of such size as to be measurable with the microscope. On examining the color and size of the particles and tabulating the results obtained, it was found that a well defined relationship existed. After one treatment particles of 0.085 micron diam, were obtained. No light was transmitted while a very little bright blue light was scattered. Another treatment increased the diam, to 0.113 micron and the transmitted light was yellow while the scattered light was blue. The third treatment gave a diam, of 0.15 micron with a brownish yellow transmitted light and a bluish scattered light. The fourth treatment gave a diam, of 0.19 micron and the transmitted and scattered light was gray. A similar progressive change was observed in other series of expts. No relationship could be established between the color of the particles and their distance apart as suggested by Zsigmondy.

W. B. Holmes.

Development of Plates Exposed thru the Glass Side. E. COUSIN. Bull. soc. franc. phot., 2, 244-6.—The development of plates exposed with the film side next to negative is often unsatisfactory especially with negatives showing great contrast. The surface of the film is highly developed while the interior is only slightly. This can be avoided somewhat by soaking the plates for some time in H<sub>2</sub>O before development. The author recommends exposing the plate on the under side thru the glass. The interior of the film then receives a larger development. This is especially advantageous with old plates which are apt to be somewhat fogged.

W. B. HOLMES.

Auto-acetinometer. C. Gravier. Bull. soc. franc. phot., 2, 242.—This auto-acetinometer consists of 2 tubes, one of which just fits inside of the other. The outer tube holds the sensitive prep. and is covered with a jacket, perforated to admit the light. It can be placed over white paper for reflected, and over glass for direct, light. The inner tube serves as an ocular for examining the sensitive prep.

W. B. H.

Contrast Screen Reenforcers in Color Photography. L. Benoist. Bull. soc. franc. phot., 2, 229-34.—White glazed paper in contact with the sensitive film is preferable to the black surface ordinarily used. There is no diffusion and the clearness, brilliancy and rendering of colors is as good with an increased luminosity in the ratio of 60-100. The length of exposure is cut from 16-10 seconds. W. B. Holmes,

The Photographic Reproduction of Documents by means of Reflection. "Cataphotography." G. DE FONTENAY. Compt. rend., 152, 1055-7.—By placing a plate in a holder with the sensitized side inside, laying the document face down on this, clamping, and exposing to the light, a negative (cataphototype) is obtained by reflection. Monochromatic red light gives the best results, but green or yellow light may also be used. Rigorously identical copies can thus be obtained very simply. The plate-holder may even be dispensed with if the room is darkened for a moment, the plate held over the document by hand, and a few matches struck, or a pocket-lamp flashed. Detailed directions are given.

M. Heidelberger.

Photographs in Changeable Colors. ESTANAVE. Compt. rend., 152, 1158-9.— The author had previously shown how, by a wire framework of horizontal lines, the subject of a photograph could be made to appear different at different angles. Now, by using a framework of colored horizontal wires it is possible to view the same subject in different colors at different angles. To do this, a modification of the Joly tri-color screen process is used, in which the screen is placed a little in front of the plate instead of directly on it. By placing the developed plate, with image reversed, in its original position relative to the screen and tilting at different angles, the subject is seen in different colors.

M. H.

Photography with Invisible Rays. R. W. Wood. Baltimore. Chem. Ztg., 34, 954.—Filters may be prepared from 2 Co-glass plates and a satd. soln. of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> or with certain dyes which only allow rays of 7000-7600 Å. units to pass. Photographic plates extremely sensitive to red are being used and also spectrum plates of Wratten and Wainwright or Cramer Co. With these the slightest trace of fog in the atm. can be detected for which reason this sort of work may become of importance in meteorology. Results with ultraviolet light 3100-3250 Å. units showed that a quartz lens, of which one side was heavily Ag plated and was opaque for visible light, was transparent to ultraviolet light and showed only 4% reflection by means of the Ag plating which is less than that of glass for invisible light. The author considers that this method will be applicable to the study of the effect of ultraviolet light on plants, butterflies, etc.

New Results on Toning Silver Bromide Pictures. ERMEN AND GAMBEL. Chem. Ztg., 34, 956.—In place of the alkali sulfides usually used to produce the brown sepia color other sulfides were successfully tried. The sulfides of As, Sb, and Sn and mixtures with or without the addition of alkali sulfides gave good results. Schlippe's salt (Na sulfoantimonate) applied to AgI pictures gave an especially rich brown color.

E. J. WITZEMANN.

Spectrography in Ultraviolet Light. ROBERT CHABOSEAU. Chem. Ztg., 34, 955.—For observations in the ultraviolet a series of modifications of fluorescing oculars have been previously proposed (Soret, Schön, Hartley, Huntington, etc.), but they only permit of observing part of the ultraviolet field at one time. In order to study the whole field the author used fluorescing and easily flexible plates, which after many expts. were finally best prepared by pouring a gelatin soln. of aesculin or uramin on flexible plates of glass, celluloid or mica. Still better results were obtained when the aesculin was followed by a layer of the uramin prep.

E. J. WITZEMANN.

Portrait Painting in its Relation to Photography. A. MEKLER. Breslau. Phot. Chronik, 1911, 360-2, 367-9.

J. J. M.

Action of Acids and Salts on Gelatin (PROCTER). 29.

MEBES: Farbenphotographie und Farbrasterplatten. Bunzlau: L. Fernbach. 8°, 301 pp., 4 M.

Brit., 16,421, July 9, 1910. A. HERZKA, 15 Gabelsbergerstrasse, Dresden-A, Saxony, Ger. In the manuf. of photographic plates without halation with the use of a colored gelatin coating sol. in H<sub>2</sub>O, for the emulsion support, the coating is distributed on both surfaces of the emulsion support, so that it can be kept so thin that it can be rendered neutral as regards the emulsion, by hardening, and prevented from giving off its dye to the latter, so that the emulsion retains its sensitiveness to light.

#### 6. INORGANIC CHEMISTRY.

#### H. I. SCHLESINGER.

Behavior of Zirconium Oxide toward Hydrofluoric Acid. E. WEDEKIND. Univ. of Strassburg. Ber., 44, 1753-5.—Thru repeated fuming with HF in presence of but little dil. H<sub>2</sub>SO<sub>4</sub>, ZrO<sub>2</sub> is volatilized almost completely. SiO<sub>2</sub> can be removed without loss of ZrO<sub>2</sub> if at least 20 wts. of conc. H<sub>2</sub>SO<sub>4</sub> and not over 45 wts. of HF are used. Direct sepn. in minerals was unsatisfactory. ZrSiO<sub>4</sub> (hyacinth) after 4 treatments gave const. wt., with loss of 19.13% SiO<sub>2</sub> instead of 33.9%. A. R. MIDDLETON.

The Deposition of Metals from Aqueous Solutions of their Salts by Hydrogen at High Temperature and Pressure. II. V. IPATEV AND V. VERKHOVSKII. Artillery Acad., St. Petersburg. Ber., 44, 1755-8; cf. C. A., 4, 7.—In the reaction, CuSO<sub>4</sub> + H<sub>2</sub> = Cu + H<sub>2</sub>SO<sub>4</sub>, the formation of basic salt and Cu<sub>2</sub>O are side reactions and not phases of the main reaction. Neither is formed if sufficient H<sub>2</sub>SO<sub>4</sub> is present. The formation of CuSO<sub>4</sub>·2Cu(OH)<sub>2</sub> is due to hydrolysis. From N CuSO<sub>4</sub> soln. about 4% of the Cu seps. in air at 100° in less than 7 days; the same amt. of ppt. is formed at lower temps. in times up to 4 weeks. Cu<sub>2</sub>O is formed only in presence of H and above a certain temp. At 50° and 700 atm. no Cu<sub>2</sub>O formed in 7 days; at 60° and 600 atm. Cu<sub>2</sub>O was formed after 2 days. Apparently an equil. exists between the amt. of Cu which seps. as metal and that which goes back into soln. from the basic salt and oxide. The data on this point are to be redetd. with a modified app. to permit of stirring.

A. R. MIDDLETON.

Tech. High-school, Carbon Ditelluride. ALFRED STOCK AND H. BLUMENTHAL. Breslau. Ber., 44, 1832-8.—On passing an arc, or strong induction sparks, between a C anode and a Te cathode under CS2, a black powder formed which at 60° went over rapidly with evolution of heat into the ord, gray form of Te. The CS, turned yellow color, had an evil odor and on evapn. left a brown residue which rapidly blackened Direct sunlight decomposed the soln. rapidly; diffused, more slowly. On distillation of the soln., the distillate retained these characteristics. Distillation in CS, vapor decomposed the solute, forming a metallic mirror on the walls of the flask. Decomp. by sunlight was incomplete and the CS, was also affected. By heating 48 hrs. at 150-200° in an evacuated sealed tube, the solute was completely decomposed to C and Te with less than 1% S. The purified residues were analyzed by heating in vacuum to 600°. A little CS2, a sublimate of TeO2 and S, a sublimate of Te, and a residue of C which retained a little Te, were sepd. and weighed. The C was burned, the sublimed TeO, being retained in the combustion tube in the final expt. The residues from decomp, of about 20 cc. of soln, weighed 70-80 mg. Results agreed well with theory for CTe2. This compd. is extremely unstable, being decomposed by sunlight into C and the black powder form of Te. It is very sol. in CS, and organic solvents. A CS, soln. at -100° deposited brilliant brown crystals which disappeared on slight warming. It has an offensive onion-like odor and acts powerfully on mucous membranes. It appears to resemble CSe, so far as can be judged from the meager de-A. R. MIDDLETON. scriptions of the latter (cf. C. A., 1, 328, 1523).

Preparation of Colloidal Silica. E. EBLER AND M. FELLNER. Univ. of Heidelburg. Ber., 44, 1915-8.—A perfectly clear and stable sol. is prepared by leading vapor of SiCl<sub>4</sub> diluted with a dry indifferent gas into dist. H<sub>2</sub>O with const. stirring. If completely freed from HCl by dialysis, it is stable for months and can be conc. to 9% SiO<sub>2</sub> without sepn. of gel. Convenient app. is described and illustrated.

A. R. MIDDLETON.

Pyrogenic Reactions of Carbon Dioxide on Carbon Disulfide and Hydrogen Sulfide. R. Meyer and S. Schuster. Tech. High School, Braunschweig. Ber., 44, 1931-44.

—The formation of CO in large amt. during the ultimate analysis of org. S compds. was ascribed by Meyer and Stadler (Ber., 17, 1576, 2075) to reduction of CO<sub>2</sub> by SO<sub>2</sub> in presence of glowing Cu. The authors passed a mixt. of these gases thru a heated tube under widely varying conditions but obtained only traces of CO or none at all. Mixts. of CO<sub>2</sub> and CS<sub>2</sub> vapor formed CO in large amt. in presence or absence of Cu. Pumice and CuO were without effect. Platinized quartz proved a very active catalyzer. Mixts. of CO<sub>2</sub> and H<sub>2</sub>S formed much CO, with some H<sub>2</sub> and S. With dry gases the reaction is CO<sub>2</sub> + CS<sub>2</sub>  $\Longrightarrow$  2CO + S<sub>2</sub>; with moist gases, CO<sub>2</sub> + CS<sub>3</sub> + H<sub>2</sub>O  $\Longrightarrow$  CO<sub>2</sub> + CO + H<sub>2</sub>S + S. CS<sub>3</sub> and H<sub>2</sub>S without CO<sub>2</sub> form neither CO nor S; the CO<sub>3</sub> is formed according to CO + S + H<sub>2</sub>O  $\Longrightarrow$  CO<sub>2</sub> + H<sub>2</sub>S. These secondary reactions occur, CO + S  $\Longrightarrow$  COS and H<sub>2</sub>S  $\Longrightarrow$  H<sub>2</sub> + S. All these substances can be present in an equil. at suitable temp. and pressure.

Allotropic Form of Silver. DRAGOMIR PALITSCH. Liége. Inst. chim. gen. Bull. sci. acad. roy. Belg., 1911, 395-415.—Pure Zn and Ag were fused in porcelain with borax and C as a flux. The grey powder obtained on treating the fusion with hot HCl is supposed to be a new allotropic form of Ag on account of the color, d., heat of amalgamation, e. m. f., and solubility.

I. K. Phelps.

Preparation of Certain Nitrides by the Reduction of Alkali Gyanides. A. C. Vournasos. Bull. soc. chim., 9, 506-12.—Finely powdered, amorphous B, previously dried at 110°, is covered with 20 parts of fragments of KCN which has been m., protected from the air, at 650° and then poured out on a marble slab; the mixture is then heated about 15 min. in a tightly covered porcelain crucible at 650°, washed free from excess of KCN with warm H<sub>2</sub>O, filtered, dried and gently ignited on a porcelain plate until all the C is burned off, and washed to remove the traces of B<sub>2</sub>O<sub>2</sub> formed during the ignition. The BN so obtained is a white powder, difficultly decomp. by hot H<sub>2</sub>O, extremely slowly by cold, H<sub>2</sub>O with formation of NH<sub>2</sub>. Al<sub>2</sub>N<sub>2</sub>, yellow amorphous powder, easily decomp. by alk. and mineral acids, is obtained in the same way. CeN and LaN, white amorphous powders easily decomp. by H<sub>2</sub>O, are prepared in the same way, altho the C cannot be burned off in the air, owing to the oxidation of the nitrides. The reaction takes place only with elements of the B group (In, Ga and Tl do not react because they m. at the temp. of the reaction).

C. A. ROUILLER.

The Fractional Precipitation of Carbonates. Roger C. Wells. J. Wash. Acad. Sci., 1, 21-4.—Dil. solns. of 2 metallic salts were pptd. by Na<sub>2</sub>CO<sub>3</sub>, enough for one metal only. The final results, detd. after the lapse of months in some cases, show the distribution of the 2 metals in the ppt. In general the sepn. is not complete. The metals may be arranged in a series, however, such that the first is pptd. to a greater extent than the next and so on, the completeness of the sepn. being greater as the metals are more removed in the series. Altho the series does not represent the order of solubility of the carbonates it does represent the pptg. power of carbonates upon metallic salts under similar conditions, which facts are applicable in geochemistry. The order is Hg, Pb, Cu, Cd, Zn, Fe, Ni, Mn, Ag, Ca, Mg. The position of Ag is remarkable.

R. C. Wells.

Metallic Thorium. Chauvenet. Chem. Ztg., 34, 940.—Th obtained by previous

workers contained only 91-92% Th. The rest consisted of Th oxide (Matignon, Delépine, etc.). The author used Li instead of Na to decompose the ThCl<sub>4</sub>. By heating ThCl<sub>4</sub> + 4Li in a Ni boat in an Fe tube to 600° a Ni alloy 88.51% Th, 11.56% Ni corresponding closely to Ti<sub>2</sub>Ni was obtained. The Ni boat was replaced by an Fe one and a quartz tube was used; a 96-96.6% Th was obtained; the 3.2% ThO<sub>2</sub> could not be removed. The Guntz method of decomposing the hydride in vacuo was tried. The ThH<sub>4</sub> was prepared by action of LiH on ThCl<sub>4</sub> at 600°. This on heating under 10 mm. gives H and 96-97% Th which is the purest yet obtained. The metal is black, does not oxidize in air nor in pure O at atm. pressure. Under pressure oxidation takes place with addition of 10% O. It burns with luminous flame in molten KClO<sub>2</sub>, combines with Cl to form ThCl<sub>4</sub> in the reaction Th + Cl<sub>4</sub> = ThCl<sub>4</sub> + approx. 339.43 cal. or 84.86 cal. for each Cl atom.

Decomposition of the Cerium Earth Double Sulfates with the Alkali Sulfates by Fusion with Charcoal. Philip E. Browning and Philip L. Blumenthal. Kent Chem. Lab., Yale Univ. Am. J. Sci., 32, 164-6.—The crude double sulfates of the Ce earths were mixed with charcoal and heated. The amts. and proportions of these substances, as well as the time and method of heating, were varied. The product on being treated with strong HCl and H<sub>2</sub>O evolved H<sub>2</sub>S abundantly, and the Ce earths went into soln., from which they were again pptd. by means of H<sub>2</sub>C<sub>2</sub>O<sub>4</sub>. The reduction of the sulfates to sulfides was not complete, altho the product was readily attacked by HCl. The best conditions for reduction seemed to be a moderate excess of charcoal with about 1 hr. heating.

Allotropic Forms and the Fusion Point of Arsenic. PIERRE JOLIBOIS. Compt. rend., 152, 1767-9.—A thermal study was made of the allotropic varieties of As, using the method already described in the author's work on P (J. ecole polytechnique, 15, 1911). The claim of Engel ( $Ann.\ phys.\ Chem.$ , 126, 218), that pptd. As is identical with that obtained in mirror form by the rapid cooling of the vapors, is confirmed. Both of these are unstable at all temps., and change by an irreversible reaction, to the gray variety in the neighborhood of 280°. The gray variety is found to be stable at all temps. up to 850°. Above 700° its vapor is yellow, but the liquid remains opaque up to 1100°. At the critical temp., however, both the liquid and the vapor are identical with the yellow, amorphous, solid form which is obtained by the rapid cooling of superheated As. The m. p. of the gray variety, as found by two methods, is somewhat indefinit. The author places it at 850°  $\pm$  10°.

Action of Nitrites and Hyposulfites on Nickel Salts (BALL). 7.

Analysis of Liquids Containing Tartaric Acid, Glycerol and Tannin (HINARD).

Determination of Amino Groups (VAN SLYKE). II.

Determination of Hydrochloric Acid in Gastric Juice (NORIEGA). II.

Determination of Phosphorus and Sulfur (KEELER). 21.

Determining Chlorides in Presence of Bromides or Iodides (NORIEGA). II.

Determining Sulfur in Coal (CUNNINGHAM). 21.

Estimating Dissolved Oxygen (Smith, Higgins). 2.

Estimation of Bisulfites (EXANGLE). 29.

12.

Estimation of Tin in Food Products (SMITH, BARTLETT). 12.

Gas Combustion Analyses (JELLER). 21.

Influences of Salts on Optical Detn. of Sucrose (Broune, Hardin). 28.

Inorganic Plant Constituents (SHEDD). 11.

New Method of Estimating Chromium. 29.

Second Spectrum of Hydrogen (CROZE). 2.

### 7. ANALYTICAL CHEMISTRY.

#### E. G. R. ARDAGH.

Advance in the Realm of Metal Analysis in 1910. TH. DÖRING. Chem. Ztg., 35, 785-6, 802-3, 821-3, 840-1.

J. J. M.

The Colorimetric Determination of Phosphoric Acid. L. POUGET AND D. CHOUCHAK. Bull. soc. chim., 9, 649-50.—The authors state briefly their method based on insolubility of strychnine phosphomolybdate (cf. C. A., 3, 1258, 2284). The method is applicable for the detn. of very small amts. of P<sub>2</sub>O<sub>3</sub> in natural waters, animal and vegetable subs., minerals, iron and steel. The reagents are prepared as follows: (A) After dissolving 95 g. MoO<sub>3</sub> and 30 g. anhyd. Na<sub>2</sub>CO<sub>3</sub> by warming in 500-600 cc. water, cool and add 200 cc. HNO<sub>3</sub> (1.4) and dil. to 1 l. (B) Dissolve by warming 2 g. neut. strychnine sulfate in about 90 cc. water, cool and dil. to 1 l. Mix 1 cc. of (B) with 10 cc. of (A) for use, filtering thru a paper that has been washed with HNO<sub>3</sub> and water. The solns. must be freshly made before using. It is suggested to have two standard solns. for comparison, one containing 0.02 mg. and the other 0.03 mg. P<sub>3</sub>O<sub>5</sub>. KH<sub>2</sub>PO<sub>4</sub> is used for standard on account of its purity, ease of prep., stability, and as it is anhydrous and not deliquescent. Dissolve 0.1918 g. of the salt in 100 cc. water, dil. 10 cc. to 1 l.; soln. contains 10 mg. P<sub>2</sub>O<sub>5</sub>.

Analysis of Calcium Cyanamide. A. STUTZER. Chem. Ztg., 35, 694.—Remarks on the methods of Monnier (cf. C. A., 5, 2793). It is pointed out that, except in very dil. solns., the method of detg. cyanamide N by adding NH<sub>4</sub>OH and AgNO<sub>3</sub>, filtering, dissolving the ppt. in HNO<sub>3</sub> and titrating the Ag with KCNS is unreliable on account of the formation of double or basic compds. containing sometimes more and sometimes less Ag than corresponds to formula for Ag cyanamide. It is recommended to use Caro's method: AgC<sub>2</sub>H<sub>2</sub>O<sub>2</sub> as a precipitant and detn. of N in ppt. Dicyanamides are detd. in the filtrate from Ag cyanamide. For the detn. of total N the Kjeldahl and salicylic acid (Förster) methods are both accurate. The author shows that the amt. of N due to the coke used in the manuf. of the carbide (called "carbon nitrogen") is very small and of no consequence for com. purposes, but would be quite considerable if Monnier's figures were correct.

Iodide Method of Copper Determinations. I. D. McClure. Mining Sci. Press, 103, 48.—Gives practically no information to one familiar with the iodide method as described in standard books.

Percy H. Walker.

Assay of Silver-bearing Gouge Ores. C. R. KEYES AND D. F. RIDDELL. Bull. Am. Inst. Mining Eng., 55, 559-69.—A critical comparison of assay metals used on certain ores from New Mexico about which there has been much controversy. The ore contained only a trace of Pb, 8.7% Fe, 25.6% Cu, 6.2% Zn, 1.0% Mn, 15.0% CaO and 10.0% S. Various methods are described and results recorded. A dead roast is not as satisfactory or as accurate as a run with a large excess of KNO<sub>2</sub> or a scorification. Excess of borax causes low results. Excessively acid or basic fluxes are hard to handle and give bad results. Ores high in Cu cannot be run by any crucible method without scorifying the Pb buttons. Direct scorification on high Cu bearing ores does not give as good results as a combination of crucible and scorification methods. The normal

or usual charge for high Cu ores (ore 0.5, PbO 1.5, NaHCO<sub>2</sub> 0.75 A. T. and KNO<sub>2</sub> 12 g., using a borax cover) gives as high results as any method on this type of ore, but does not check as well as when a large excess of Pb is used. A large excess of Pb in the charge greatly improves the buttons and renders them easily handled, also decreases the time and temp. of fusion and gives accurate results. The charge used was the normal charge with the addition of 3 A. T. PbO. The buttons were clean, soft, malleable and were scorified with 40 g. test lead. The method is quick, easy and accurate.

PERCY H. WALKER.

Compounds Produced by the Simultaneous Action of Nitrites and Hyposulfites on Nickel Salts. A Method for the Detection of Nickel in Presence of Much Cobalt. W. C. Ball. Proc. Chem. Soc., 26, 329.—When Na<sub>2</sub>S<sub>2</sub>O<sub>2</sub> is gradually added to a soln. of a mixt. of Ni(NO<sub>2</sub>), with NaNO<sub>2</sub> or KNO<sub>2</sub> the liquid assumes the color of permanganate and needle-shaped, dark purple crystals sep. The Na compd. possibly has the comp. represented by the formula: Na<sub>2</sub>NiNS<sub>2</sub>O<sub>4</sub>.4H<sub>2</sub>O. Both salts yield purple ppts. with Ba and Sr solns. Similar complex cyanides of an intense reddish purple color may be obtained by treating the above substances with KCN, and also by oxidation of ammoniacal solns. of hydroxylamine containing a Ni salt and a cyanide. They are sol. in alc. and other organic solvents and afford a ready means of detecting Ni in presence of much Cu. The compds. are all very unstable.

Percy H. Walker.

Volumetric Determination of Lead Peroxide. J. F. SACHER. Chem. Ztg., 35, 731.—It is stated that the method of Lux as usually executed gives low results, due to some PbO<sub>2</sub> being dissolved by the HNO<sub>2</sub>, but that correct results are obtained by adding the C<sub>2</sub>H<sub>2</sub>O<sub>4</sub> before the HNO<sub>2</sub>. Other methods are referred to briefly, but the Lux, as modified, is recommended as the most accurate and rapid. F. W. SMITHER.

A New Method of Determining Nitric Acid. G. ROMIJN. Z. anal. Chem., 50, 566-70.—The different methods in use with their advantages and disadvantages are briefly discussed and the following method is recommended as simple, easily executed and as enabling one to det. accurately as little as 0.1 mg. NO, ion per 1. Rub up carefully "extra fine" Zn dust with an equal amt. of kieselguhr; mix 100 g. (NH4)2SO4 with 100 cc. NH<sub>4</sub>OH (d. 0.96) and water sufficient to make 300 cc. Place 5 cc. of this soln. in a 100 cc. flask and add nitrate soln. (which should not contain more than 400 mg. NO, ion per l.) up to mark, then add about 200 mg. Zn dust mixt., stopper and shake violently. Let stand about 5 min. and filter thru a dry paper into a dry flask, rejecting the first portion and returning the filtrate until perfectly clear—the time so required ensures the completion of the reduction. In the filtrate will be NO, ions corresponding to 95% of the NO, ions that were in the soln. For larger amts. an aliquot is titrated with KMnO4. To avoid loss of N2O3, put 5 cc. of the filtrate into a 100 cc. stoppered flask, add 10 cc. 0.1 N KMnO4 and 5 cc. H<sub>2</sub>SO4 (d. 1.124), stopper the flask, shake and then let stand 15 min. Now add 5 cc. KI soln. (1: 10) and det. the liberated I with 0.1 N Na<sub>2</sub>S<sub>2</sub>O<sub>2</sub>.5H<sub>2</sub>O (1 cc. = 2.30 g. NO<sub>2</sub>). The method gives results from 2-3% too high as shown by expts. and the author states that he does not know "to what causes this excess must be attributed." F. W. SMITHER.

Determination of Lead in Lead Arsenate as Lead Chromate. C. C. McDonnella, and R. C. Roark. 'U. S. Dept. of Agr., Bur. Chem., Bull. 137, 40-2.—The usual, method of detg. Pb as PbCrO<sub>4</sub> in HOAc soln. is not applicable to Pb arsenate as this compd. is insol. in HOAc. A modification of the method has been devized as follows: Dissolve 2 g. sample in 50 cc. of HNO<sub>4</sub> (1:4) heating if necessary, cool, dil. to 200 cc., pipet 50 cc. of this soln. into a 400 cc. beaker, dil. to 300 cc., heat nearly to b., add NaOH or NH<sub>4</sub>OH to incipient pptn., then dil. HNO<sub>3</sub> (1:10) to redissolve the ppt., avoiding more than a slight excess; and then add slowly to this b. soln. 50 cc. of a hot 10% K<sub>2</sub>CrO<sub>4</sub> soln. with constant stirring. Place on steam bath about 15 mins., filter

hot collecting the ppt. in a weighed gooch and wash with b. distilled water until wash water is no longer yellow. Dry in air oven at 140-150° to const. wt. and weigh as PbCrO<sub>4</sub>. The asbestos filter mat should be thick and pressed down firmly, then washed, and dried at 140-150° to const. wt. The method is quite rapid and accurate and applicable not only to com. Pb arsenates but other compds. containing Pb as well, except in the presence of Ag, Bi, Ba, Mn, Hg, or much Fe. C. C. McDonnell.

The Dichromate-Iodide Method fo Lead. R. T. WILDER. Eng. Mining J., 92, 390.—The following method for the detn. of Pb in ores is a modification of Low's scheme and gives good results. 0.5 g. of ore is treated as is usual in the molybdate method up to the point where the washed sulfate is obtained on the filter paper. The paper and ppt. are placed in a 200 cc. grad. flask, and a few g. of AcONa, I cc. of AcOH and about 50 cc. of H<sub>2</sub>O are added. The flask is heated until the PbSO<sub>4</sub> is in soln. An excess of dichromate soln. (3.558 g. per 1.) is now run in and after mixing by shaking the contents are made up to the mark with H<sub>2</sub>O. Shake, filter thru 3 dry filters folded together, take 100 cc. of the filtrate, add a few cc. of H<sub>2</sub>SO<sub>4</sub> and enough KI to react with excess of chromic acid and titrate with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (9.150 g. per 1) in the usual manner. If CaSO<sub>4</sub> or basic ferric sulfate are present in the ore they must be removed or a method used where they cannot interfere.

The Determination of Gold and Silver in Black Copper. H. NISSENSON. Z. angew. Chem., 23, 968.—The combined dry and wet method has been simplified by the author as follows: 8 samples of 12.5 g. each are weighed out in clay crucibles and each mixed with 20 g. of a black flux made by heating together 1 part of KNO<sub>3</sub>, 3 parts of argols, cooling, pulverizing and mixing with 2½ times their mass of soda and borax. 25 g. of litharge and about 60 g. of a mixt. containing 7 parts soda, 6.5 parts borax and 1 part of argols are now added and the mixts. melted in a blast furnace. After this fusion 200 g. of Pb are added and the whole heated strongly for 5 min. The fused mixts. are then poured out, cooled, divided into 5 parts and the Ag and Au sepd. and detd. according to the usual wet method.

G. W. STRATTON.

A New Quick Method for the Determination of Nitrate Nitrogen. A. QUARTAROLI. Viterbo. Chem. Lab. of the Tech. Inst. Staz. sper. agrar. ital., 44, 157-64; thru Chem. Zentr., 1911, II, 49.—The method is based upon the observed fact, that nitrates react with anhydrous  $CO_2H_2$ , according to the following equation:  $2KNO_3 + 6H.COOH = N_3O + 4CO_3 + 5H_2O + 2H.COOK$ . The test is carried out as follows: 0.1-0.3 g. of the nitrate is heated with 5 cc.  $CO_2H_2$  and the gases evolved are collected over Hg in a eudiometer tube of 200-250 cc. capacity. After reading the vol. of the gases, 2 cc. conc. KOH soln. is added, the tube shaken, and the vol. again read. The first reading equals  $CO_2 + N_3O$ , the second,  $N_2O$ . The values are computed to 0° and 760 mm. pressure. In 2 tables the results for  $CO_2 + N_3O$  and for  $N_3O$  as calc. and as found are given and in a third table a comparison is made between the N thus found and the results according to Schulze-Tiemann. They agree satisfactorily.

J. A. LECLERC.

Parting Platinum-Gold-Silver Bullion. Anon. Eng. Mining J., 92, 259.—Ag is added if necessary to bring up the ratio of this metal to 75% in the alloy. The metal is then cast and rolled into a very thin sheet. Strong H<sub>2</sub>SO<sub>4</sub> with heating is used to effect parting, 2–3 oz. acid per oz. metal being used. After action is complete the soln. is poured into about 9 vols. H<sub>2</sub>O and the residue washed by decantation. Ag is pptd. with wire or sheet Cu. The residue is digested in aqua regia and any AgCl formed is filtered off. NH<sub>4</sub>OH is added till the soln. is nearly neutral, the Pt is pptd. with NH<sub>4</sub>Cl and is filtered off and ignited. In the subsequent filtrate Au is pptd. with FeSO<sub>4</sub>, filtered off and melted under borax.

New Reaction for the Stannous Ion. JULIO DE G. CANANCIO. Anales soc. espans. fis. quim., 9, 177-8.—When KSCN and a reducing agent are added to an HCl soln. of molybdic acid or a molybdate a carmine-red color is produced. This color reaction is made use of in the present test. The reagent consists of 15 g. NH<sub>4</sub>SCN (preferable to KSCN) in 250 cc. H<sub>2</sub>O to which is added 1 cc. of a soln. of 1 g. NH<sub>4</sub> molybdate in 10 cc. HCl (sp. gr. 1.19). The reagent is best added slowly to the soln. to be tested and without heating. 0.0001 g. SnCl<sub>2</sub> in 1 cc. can thus be detected. SbCl<sub>3</sub> gives the color reaction only slowly and Sn" can be detected in presence of Sb". H<sub>2</sub>S and other reducing agents give this color reaction with the above reagent. H. S. Paine.

The Determination of Vanadium in Steel and Iron. B. O. CRITES. Textor Lab., Cleveland, O. J. Ind. Eng. Chem., 3, 574-7.—The limits of accuracy of the methods of Johnson, Blair and others for the detn. of V in steels are indicated in the tabulated results obtained in comparative analyses.

H. M. LANCASTER.

A Simple Method for the Detection and Estimation of Nitric and Nitrous Acids in Water. J. Tillmans and W. Sutthoff. Städt. Hyg. Inst., Frankfurt a/M. Z. anal. Chem., 50, 473-95.—The diphenylamine reaction for nitrates in waters as hitherto applied, gives irregular results. This is due, in part to the lack of sufficient chloride. By adding NaCl as reagent to the reacting mixture, the degree of accuracy uniformly obtainable may be increased to 0.1 mg. HNO<sub>2</sub> per l. Comparative results obtained in the examination of solns. containing definit amts. of nitrate in high dilution show that this process compares favorably with those of Noll, Ulsch, and Schulze-Tiemann. Nitrites if present react similarly to nitrates, and a combined detn. may be made. The method as outlined requires no special app., is rapid and employs only a small amt. of sample.

The Loss of Carbon during Solution of Steel in Potassium Cupric Chloride. H. ISHAM. Univ. III., Urbana. J. Ind. Eng. Chem., 3, 577-9.—From expts. outlined, the apparent loss of C during soln. of steel in K<sub>2</sub>CuCl<sub>4</sub> at ordinary temp. (C. A., 2, 3210), is due to gaseous compds. of C present in the reagent and not to the formation of such from the C in the steel. There is no appreciable loss of C in steel.

H. M. LANCASTER.

The Colorimetric Determination of Mercury in Urin. A. HEINZELMANN. Chem. Ztg., 35, 721-2.—The author has made a critical study of the method of Schumacher and Jung (Z. anal. Chem., 41, 461) and recommends that it be carried out as follows: 500 cc. urin in a l. flask are treated with 5 g. KClO, and 50 cc. HCl (1.12) and heated till the soln. turns from dark red to bright yellow, cooled to about 70° and 10 g. of pure Zn filings added, let stand about 12 hrs. (over-night) and the urin decanted from the Zn, which is washed well with water. Now add to the Zn (containing the Hg) in the flask 50 cc. HCl (1.18), warming after some time, if necessary, to make soln. complete; add 20 g. KOAc, pour into a 100 cc. Hehner cylinder and make up to 90 cc. with water and then to 100 cc. with satd. H<sub>2</sub>S soln., mixing with a rod bent to form a ring at one end. In the meantime the same amts. of the Zn, HCl (1.18) and KOAc are used for making a standard soln., adding with pipet 5 or 10 cc. of HgCl, soln. ( = 1 or 2 mg. Hg), transfer to a Hehner cylinder and treat as above and compare colorimetrically as usual. The H<sub>2</sub>S should be added to the standard and unknown at the same time and all conditions as to the amts. of reagents, etc., adhered to in each case. The results reported on amt. of Hg recovered from a known mixt. show excellent agreement.

F. W. SMITHER.

Remarks on Hans Friedenthal's Paper, "Quantitative Chemical Analysis of Mixtures by the Use of Differences in their Specific Gravity." M. A. RAKUZIN. Ber., 44, 1676.—Centrifuging at the rate of 3000 revolutions per min., as proposed by Frieden-

thal (C. A., 5, 2048) produces changes in the optical and other properties of petroleums. The author's expts. on this subject were published in Russian, in 1908. The specimens of oil employed were covered with collodium, to prevent the loss of the more volatil constituents. After being centrifuged the d. diminished 0.002-0.003 and the "carbonization constant" rose about 30%. The centrifugal force must be included as a geomechanical factor in the formation of petroleum. J. BISHOP TINGLE.

Quick Method for Determining Ether Extract in Dry Powdered Substances. A. E. LEACH AND R. S. HILTNER. U. S. Dept. of Agr., Bur, of Chem., Bull. 137, 85.-Weigh 2 g. of the substance upon a watch glass and transfer to a 100 cc. beaker. Treat with 20 cc. of ordinary ether and shake continuously by rotating the beaker, taking care to avoid loss. Let stand for a few minutes, then transfer rapidly (by the aid of a stream of ether) to a quick-acting filter in a funnel placed in the neck of a tared 100 cc. Erlenmeyer flask. Wash with ether till the filtrate amounts to about 75 cc., avoiding the formation of a layer of ether extract at the top of the filter paper. Allow the extract to remain at 40-50° until all of the ether has evapd. (17-18 hrs.), cool and weigh.

W. D. BIGELOW.

The Detection of Traces of Hydrogen Cyanide. G. Druce Lander and A. E. WALDEN. Analyst, 36, 266.—For quantities of HCN less than 0.00002 g. per cc.. Add to the soln. 2 drops of 10% NaOH and evap. in a test tube nearly to dryness; cool, add 1 drop of 2% FeSO, and leave in the cold for 10-15 min.; add 2-3 drops of strong HCl, warm gently and cool. The soln. is blue-green and on careful diln. shows a blue color. By this method 0.00001 g. can be recognized with certainty and definit positive results are obtainable with 0.000002 g. The picric acid method is a very general reduction method, the red coloration being given by any substance that will reduce picric acid in alkaline soln. When the presence of HCN has been shown in the absence of other reducing agents the picric acid method is valuable in detg. the HCN quantitatively. C. W. CLARK.

EMICH, F.: Lehrbuch der Mikrochemie. Wiesbaden: J. F. Bergmann. 8°, 212 pp., 6.65 M.

FURMAN, H. V. AND PARDOE, W. D.: Manual of Practical Assaying. Seventh edition. New York: Wiley. 8°, 530 pp., \$3.00.

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SMITH, J. R.: Modern Assaying. Lippincott. 145 pp., \$1.50.

TREADWELL, F. P.: Analytical Chemistry. Vol. II. Quantitative Analysis. Translated by W. T. Hall. Third edition. New York: Wiley. 900 pp., \$4.00.

## 8. MINERALOGICAL AND GEOLOGICAL CHEMISTRY.

#### R. C. WELLS.

A New Locality of Pyrrhotite Crystals and their Pseudomorphs. C. H. Smyth, Jr. Am. J. Soi., 32, 156-60.—In a calcareous sandstone near Oriskany; N. Y., were found small prismatic crysts. of pyrrhotite and also thin scales of the same considerably weathered. The pyrrhotite rested on calcite when the latter was present, otherwise on dolomite or lastly on quartz if the pyrrhotite crysts. were large. This is a rather exceptional association. The pseudomorphs were limonite and pyrite.

Ferritungstite, a New Mineral. W. T. Schaller. J. Wash. Acad. Sci., 1, 24;

Am. J. Sci., 32, 161-2.—A mineral from Washington crystallizing in hexagonal plates gave WO<sub>2</sub> 37.1 and 35.8, Fe<sub>2</sub>O<sub>2</sub> 26.6 and 27.3, H<sub>2</sub>O (ign.) 18.6 and 20.9, insol. 14.7, from which the formula Fe<sub>2</sub>O<sub>2</sub>.WO<sub>2</sub>.6H<sub>2</sub>O is deduced. The name ferritungstate is assigned to this hydrated ferric tungstate.

R. C. Wells.

Hinsdalite, a New Mineral. E. S. LARSEN AND W. T. SCHALLER. U. S. Geol. Survey. I. Wash. Acad. Sci., 1, 25-6.—Hinsdalite was first collected near Lake City, Hinsdale County, Colorado where it is abundant as an original vein mineral associated with quartz and a little pyrite, galena, tetrahedrite, and barite. It occurs in bands an inch or so across and as crystals embedded in granular quartz. The crystals are either rhombohedrons, resembling cubes, or pseudo-hexagonal tablets; perfect basal cleavage; hardness about 5; luster vitreous to greasy. The fresh mineral is pale greenish, but much of the material is dark gray from inclusions. The streak is colorless. The indices of refraction are somewhat variable but the values for the principal zones are about  $\alpha = 1.670$ ,  $\beta = 1.671$ ,  $\gamma = 1.688$ . Sections normal to the cleavage show parallel extinction. 2E is usually about 32°. Basal sections of some of the crystals are divided into 6 radial segments and the plane of the optic axis in each segment is normal to the pseudo-hexagonal prism edge. The following analysis was made on fresh, light gray crystals: PbO 31.75, SrO 3.11, Al<sub>2</sub>O<sub>2</sub> 26.47, SO<sub>2</sub> 14.13, P<sub>2</sub>O<sub>3</sub> 14.50, H<sub>2</sub>O 10.25, CaO, MgO, Na<sub>2</sub>O, K<sub>2</sub>O traces. Density = 3.64. Formula, 2(Pb, Sr)0.3Al<sub>2</sub>O<sub>2</sub>. P<sub>2</sub>O<sub>3</sub>, 2SO<sub>3</sub>, 6H<sub>2</sub>O. Infusible, losing H<sub>2</sub>O wholly only at about 500°.

Beaverite, a New Mineral. B. S. BUTLER AND W. T. SCHALLER. U. S. Geol. Survey. J. Wash. Acad. Sci., 1, 26-7.—Near Frisco, Beaver Co., Utah, where primary ores have been extensively altered to a depth of 600 ft. by descending solns., and this alteration has taken place to a lesser extent to a much greater depth, the following secondary minerals have been recognized: Anglesite, cerusite plumbojarosite, jarosite, beaverite, linarite, bindheimite (?), smithsonite, calamine, goslarite, covellite, chalcocite, brochantite, malachite, azurite, chrysocolla, chalcanthite, cerargyrite, sulfur, chalcedony, kaolinite, gypsum, hydrous oxide of iron and manganese, and alunite (?). Beaverite commonly occurs mixed with other secondary minerals but occasionally is found pure in small, friable masses. It is canary-yellow and crystallizes in 6-sided plates too small for measurement of the angle or detn. of the optical character. Minute undetd. inclusions are present in nearly every crystal. The mineral is sol. in b. HCl, leaving an insol. siliceous residue. The analysis follows: Insoluble 10.05, CuO 9.70, PbO 29.44, Fe<sub>2</sub>O<sub>2</sub> 17.28, Al<sub>2</sub>O<sub>2</sub> 3.64, SO<sub>2</sub> 21.32, H<sub>2</sub>O<sub>3</sub> 9.02, sum 100.45. The ratios agree well with the formula CuO.PbO.Fe<sub>2</sub>O<sub>2.2</sub>SO<sub>2.4</sub>H<sub>2</sub>O. The water is all constitutional as none was driven off below 250°. There is no known mineral to which beaverite is related so that at present it stands as an isolated member of the sulfate group.

R. C. WELLS.

Eglestonite from San Mateo Co., California. A. F. ROGERS. Am. J. Sci., 32, 48-50.—In a serpentine rock, associated with cinnabar and calomel, about 5 miles west of Palo Alto, were found minute crysts. and also crusts of eglestonite. Four out of the 7 forms described by Schaller (U. S. Geol. Survey, Bull. 405, 148) were recognized. Analysis gave Hg 88.00, Cl 7.43%, SO<sub>2</sub> none, which with the optical tests, proves the identity of the substance.

R. C. Wells.

The Stability Ranges of Minerals with Special Reference to the Geologic Thermometer Scale. Fred E. Wright. J. Wash. Acad. Sci., 1, 45.—The physico-chemical systems represented by rocks and the geologic forces which act upon these systems are often so complex that it is not possible at present to solve completely the problems which they present and to state definitly the behavior of any particular rock or magma under the different conditions of temp. and pressure which may arise. It is important, therefore, that limits of temp. and pressure be established, beyond which any given

system or any component of the system is no longer stable but undergoes change. These limits mark points on the geologic thermometer scale or pressure gage and place definit boundaries to the region in which the observed reactions have taken place. At these points an abrupt shift in the equil. of the system occurs and produces profound changes in the system. The most important thermometric points which may be of service in this connection are: (a) Enantiotropic inversion temps. of minerals, as that of  $\alpha$ -quartz into  $\beta$ -quartz; (b) temp. limits beyond which monotropic or isomeric forms cannot exist, as the temp. 450°, above which marcasite passes at atm. pressure into pyrite; (c) stability ranges of enantiotropic forms and of minerals which dissociate or decompose at higher temps, as the dissociation temps, for different pressures, or calcite and other carbonates or of certain of the garnets and many other contact minerals; (d) m. temps. of stable minerals and of eutectics; (e) temps. beyond which any physical property acquires a permanent set and by virtue of internal friction or other cause does not revert to its original state on cooling. In applying these data of lab. expt. to geologic problems, it is important to note the conditions under which the data were obtained and the effect which other conditions of pressure or soln. may have exerted on the observed points of equil. change. R. C. WELLS.

The Amount and Vertical Distribution of Water Vapor on Clear Days. W. J. Humphreys. J. Wash. Acad. Sci., 1, 1.—The amt. of water vapor rapidly decreases with elevation; but whatever the humidity a first approximation to the av. total amt. of water vapor above any given level is expressed by the equation, d = 2e, in which d is the thickness in mm. of the equiv. water layer, and e the partial pressure of the water vapor, at the given level, in mm. of Hg.

R. C. Wells.

Note on the Composition of Sea Water. F. W. CLARKE AND GEORGE STRIGER. J. Wash. Acad. Sci., 1, 4.—Altho the water of the ocean varies widely in salinity, from less than 1% in the Baltic to 5% in the Red Sea, the comp. of its saline matter is curiously constant. Data are so far lacking, however, with regard to the waters of American coasts, except for a single group of analyses, by A. S. Wheeler, of water from near Beaufort, N. C. In order to remedy this deficiency, a sample of water from near Loggerhead Key in the Gulf of Mexico was analyzed. The sp. gr. was 1.02434, at 25°, and the total salinity 3.63516%. This is slightly above the mean salinity of the ocean, 3.5%. The % comp. of the saline matter is given below: Cl 55.24, Br 0.17, SO<sub>4</sub> 7.54, CO<sub>2</sub> 0.34, Na 30.80, K 1.10, Ca 1.22, Mg 3.59. R. C. Wells.

Origin of Hydrocarbons. G. F. BECKER. U. S. Geol. Survey. Eng. Min. J., 92, 117; cf. C. A., 5, 1836.

ROBERT KANN.

The Problem of Keromorphy in the Vegetation of the Carboniferous Period. A. DACHNOWSKI. Am. J. Sci., 32, 33-9.—The author does not believe it necessary to postulate a warmer moister atmosphere more heavily charged with CO<sub>2</sub> than at present to account for the formation of the coal measures. The conditions were probably similar to those in bogs and marshes to-day. These conditions are only now becoming known and include hitherto neglected variables. In peat soils the oxidation zone extends scarcely more than 2 ft. from the surface, the water table being at a high level. Yet this water becomes unavailable to plants on account of the presence of various decomp. products, fungi and the activity of low organisms and bacteria. The latter include diastatic, inverting, proteolytic and cytohydrolytic. On account of toxic effects of the decomp. products and of the bacteria, much of the flora of the coal measures is really that of a dry region altho developed in a wet environment.

R. C. WELLS.

The Chemical Composition of the Bowlder Loams in the Netherlands Diluvium, with Special Consideration of the Silicate Disintegration. G. H. LEOPOLD. Reichs-

landwirtsch. Versuchsstation, Holland. van Bemmelen Gedenkboek, 74-87; thru Chem. Zentr., 1911, I, 1153.

F. W. SMITHER.

Sulfur Deposits near Soda Springs, Idaho. R. W. RICHARDS AND J. H. BRIDGES. U. S. Geol. Survey, Bull. 470-J, 8 pp.—In sulfur canyon S is found in a breccia of tuff, limestone and quartz. Gypsum is also present in small amt. The source of the S, however, is believed to be some near-by volcanic center. The S is found in small pyramidal crystals lining cavities, in masses in the interstices of the breccia and as small stalactites whose fracture changes on exposure to the air from a canary-yellow to a submetallic gray.

R. C. Wells.

Geologic Atlas of the United States, Foxburg-Clarion Folio, Pennsylvania. E. W. Shaw and M. J. Munn. U. S. Geol. Survey, 17 pp., maps.—Describes oil formations and gives analyses of coal, oil, and iron ore.

R. C. Wells.

Oil and Gas Fields of the Carnegie Quadrangle, Penn. M. J. Munn. U. S. Geol. Survey, Bull. 456, 99 pp., maps.—Stratigraphic. R. C. Wells.

Deposition of Metals (IPATEV, VERKHOVSKII). 6. Mineral Flotation (MICKLE). 9.

Oil Fields of Sakhalin (BEHAGHEL). 22.

Radioactivity of Igneous Rocks (FLETCHER). 3.

Weathering of Silicates (STREMME). 15.

CIRKEL, F.: Chrysotile Asbestos: Its Occurrence, Exploitation, Milling and Uses. Canada Dept. of Mines, Publication No. 69.

DORLTER, C: Handbuch der Mineralchemie. Dresden: T. Steinkopff. 8°, 6.50 M.

Brit., 17,240, July 20, 1910. K. Mikimoto, 3 Shichome-Ginza, Tokyo, Japan. Causing oysters to produce pearls. See C. A., 5, 2058.

# 9. METALLURGY.

# WM. BRADY, W. T. HALL.

Continuous Decantation. DONALD F. IRWIN. Mining Sci. Press, 103, 102-3.—
A description with diagrams of 2 types of plant for slime treatment, the fundamental idea in both being successive decantation, thickening, and increasing diln., thru a tandem set of 3 Dorr thickeners.

E. J. Crane.

Copper for Casting. Anon. Mining Sci. Press, 103, 141.—In selecting Cu for casting it is important to know the brand because of the definit information as regards its sp. properties that is then obtainable. The greater the elec. cond. the greater the purity; this is especially true in regard to As. With 0.03-0.80% As present, relative cond. is decreased from 100 to 40. The impurities most found besides CuO are As, Sb, Fe, S, Bi, Se, and Te. 99.9% purity is "very satisfactory" for casting.

E. J. CRANE.

Electroplating Battery Plates. J. D. HUBBARD. Mining Sci. Press, 103, 135-6.

—The chem. comp. of the H<sub>2</sub>O, quality of Hg used, size and condition of the pulp passing over, and the particular material used in dressing the plates, all have a tendency to shorten the life of battery plates. "Brushing up" with strong cyanide soln. should not be practiced. KCN, when used intermittently on amalgamated Cu plates, will

gradually form double cyanides of Hg and Cu, causing the Cu to form verdigris, which will in turn lift amalgam and even Ag plating and cause it to scale off the plate. Hard, oxidized Cu plates may be made fit for use again by scraping them well, heating to dull redness, plunging into cold H<sub>2</sub>O to anneal them, and hammering out irregularities with wooden block and mallet. This process is often best followed by Ag plating. The plates annealed as above should be scoured with fine sand and the Ag plating continued till there has been a deposition of 2.5 oz. Ag per sq. ft. of surface. The strength of the Ag-plating AgCN soln. should be maintained between 1 and 2%, never below 1%. It is best prepared by dissolving AgNO<sub>3</sub>, pptg. with NaCl, and dissolving the AgCl in KCN soln. Only pure Ag anodes, dist. H<sub>2</sub>O, and high grade KCN should be used. Free C<sub>2</sub>N<sub>2</sub> should be 1% in excess in the plating soln. Large wires must be used from the dynamo.

Valuation of Ores. IV. A. RZEHULKA. Z. angew. Chem., 24, 444-7; cf. C. A., 4, 1971.—This paper deals with Sn and W ores. It describes their occurrence in veins and alluvial deposits and goes into detail as to the important districts, laying special emphasis on influence of locality and surrounding rocks on the quality of these ores. Methods of treatment are described with important comments. A universal formula for valuation of ores, follows: (For tim) W = (P Tin)/100 - X where W = value of the ores in marks, P = av. Sn content in %, Tin = current market price of Sn in marks and X = deduction for loss in mining and smelting operations. Statistical details of production of Sn with prevailing prices for the years 1900–1909 are given. Details are given of the relative values of W ores. The world's annual production of W ore is estd. 3300 tons and valued at  $1^1/2$  million dollars. J. S. Goldbaum.

Output of Platinum, Iridium, Palladium and Rhodium. Anon. Bross World, 7, 292-3.—Figures taken from advance chapters of U. S. Geological Survey publications.

ROBERT KANN.

Nickel Steel Castings. S. Kern. Chem. News, 103, 169.—The crucible steel at the foundry of New Admirality is prepared in coke furnaces. The charge per crucible is 100 lbs. and consists of 10.53% puddled Fe (0.07% C, Mn trace), 43% of punchings, 44% of rivet steel in ends and chippings, 1% of Ni and 0.15% of 80% ferromanganese. Before withdrawing the crucibles 0.6% ferrosilicon and 0.65% silicospiegel is added to the melted steel. Before pouring, each crucible receives 0.07% Al. The resulting steel contains C 0.43%, Ni 0.72, Mn 0.60, Si 0.37. After annealing the steel, mechanical tests show about 50 tons per sq. in. of absolute resistance and 10-12% of elongation in 2 in.

The Value of Coarse Concentration. A. L. STUART. Eng. Mining J., 92, 243.—Discussion of Adkinson's article (C. A., 5, 2800).

ROBERT KANN.

Cyaniding Concentrate at Waihi. Anon. Eng. Mining J., 92, 248.—Grinding is done in CN soln. About 22% Au and 28% Ag are recovered by conc. After the addition of lime and grinding the concentrates are agitated by air for 8-10 days in cone-bottom vats. 0.25 lb. NaCN is used per ton of ore treated. Au and Ag are recovered by Zn shavings.

ROBERT KANN.

Economic Features of Porcupine Ores. W. L. Fleming. Eng. Mining J., 92, 253-6.

ROBERT KANN.

Efficiency of Fine Grinding Machinery. W. H. Urbiter. Eng. Mining J., 92, 257-9. ROBERT KANN.

The Sydvaranger Iron Mines. D. E. WOODBRIDGE. Eng. Mining J., 92, 260-4.

Over 450,000,000 tons of pure magnetite ore, averaging 35% Fe lie above sea level. The final concentrate averages: 69%, Fe; 0.015%, S; and 0.006%, P.

ROBERT KANN.

Progress in Milling on the Rand. F. H. HATCH. Eng. Mining J., 92, 265-6.

ROBERT KANN.

Experiments on Mineral Flotation. K. A. MICKLE. Eng. Mining J., 92, 307-10; Proc. Roy. Soc. Victoria, 23, March, 1911.—A series of lab. expts. are described on the buoyancy of a wide variety of minerals. The conclusions show that: all minerals absorb gases. Sulfides often absorb CO<sub>2</sub> naturally. Most of the minerals behave in cold dil. acid solns. as in H<sub>2</sub>O. In hot acids their buoyancy is markedly less. Minerals immersed in an alkaline soln. tend to lose their absorbed gases and hence buoyant property. Finely divided particles when floated quickly break thru the surface and sink. In general, heat aids flotation. Gases collect more readily on oiled than on unoiled particles and hence increase their tendency to float. Selective flotation of sulfides and gang can be accomplished by oiling the material and introducing it into a dil. acid bath. The oil tends to leave the gang and become attached to the sulfide particles. Flotation may be accomplished in alkaline solns. when oil is used, but the action is non-selective.

Smelter Settlements and Contracts. I. P. E. BARBOUR. Eng. Mining J., 92, 314-8.

ROBERT KANN.

The Porcupine Gold Area. A. G. Burrows. Ontario Bur. Mines, 1911. 20th Annual rept. Eng. Mining J., 92, 348.

ROBERT KANN.

Trojan Cyanide Mill, Black Hills. J. SIMMONS. Eng. Mining J., 92, 357-8.— A unique equipment in the Pachuca tanks is an improvement over the usual arrangement. A series of sheet-steel forms, each the frustrum of a cone, is attached to a frame so that the top of each cone is a couple of ins. above level of the bottom of the one next above. These extend from the top to the bottom of the tank, permitting a perfect agitation by compressed air, no matter what the depth of material in the tank. The av. time of agitation of slime is 5-6 hrs.

ROBERT KANN.

Smelter Settlements and Contracts. II. P. E. BARBOUR. Eng. Mining J., 92, 364-6. ROBERT KANN.

The Saving of Fine Placer Gold. F. H. HAZARD. Eng. Mining J., 92, 394-6.—
The problem of saving fine placer Au has not received sufficient attention. Any process whereby black sands may be collected for independent treatment will promote economy.

ROBERT KANN.

Zinc Dust Precipitation. C. W. MERRILL. Eng. Mining J., 92, 407—One of the noteworthy features in the development of recent CN practice is the increasing use of Zn dust as a precipitant for the precious metal solns. As compared with shavings method, the first cost of Zn dust is from 20-25% less. In the new Zn-dust process the reduction is accomplished by bringing the precipitant and the solns. into contact with the entire exclusion of air and maintaining this condition until the ppt. has been filter-pressed. Resolution of the pptd. precious metals by oxidation is thus prevented.

ROBERT KANN.

Tin Mining and Milling in Bolivia. III. M. ORMAS. Eng. Mining J., 92, 41 f-3.

ROBERT KANN.

Cyanidation of Ores. W. A. HENDRYX. Met. Chem. Eng., 9, 82-5.—The ores are broken to less than 1 in. size, the product being delivered to the stamps where a weak CN soln. is employed. Amalgamation follows if coarse Au is present. The product is then passed into a dewatering sizer, the resulting fines being transferred to a combination agitator and filter. A detailed description of the agitator is given.

ROBERT KANN.

Reverberatory Copper Smelting. P. B. WILSON. Mines and Minerals, 31, 557-61.—A review. ROBERT KANN.

Cyanidation of Concentrate. E. G. Banks. *Mining Sci. Press*, 102, 142.— The concentrate at the Waihi mine, New Zealand, consists chiefly of Fe sulfide with a small proportion of Zn, Cu and Pb sulfides. It assays about 5.5 oz. Au and 65 oz. Ag per ton. The concentrate is ground in tube mills and delivered to spitzkasten, the coarse returning to the tube mills and the fine passing to dewatering boxes. Agitation by air for 8-10 days is carried out in conical bottom tall tanks, the soln. being kept at an average strength of 0.4% KCN. Consumption of NaCN averages 0.25 lb. per ton of original ore.

Treatment of Silver Ore in Guanacevi, Mexico. R. C. KLINE. Mining Sci. Press, 102, 402-5.—The ores are classified as oxidized and unoxidized, the former varying little in % metallurgical comp., but yielding vastly different % extraction under identical treatment. Oxides of Fe and Mn were present in considerable quantity, together with small amts. of Zn. The best treatment is as follows: (1) Crush in soln, so that only 2\% remains on 200 mesh. (2) Sufficient CN is added to bring the soln. up to a strength of 2.5 lb. true free cyanide per ton. (3) Agitate continuously for 30 hrs. After decantation, filter the charge. (4) Wash filter cakes, in position, with from  $1^{1}/_{2}$ -2 tons of barren soln. per ton of slime. (5) Add lime at the battery to maintain a pro ective alkalinity of 2.5 lb. per ton of soln. The unoxidized ores were made up of: (1) A clean quartz, with Ag as sulfide, and some stephanite; (2) a very clean ore, with Ag as pyrargrite; (3) a light brown ore (partly oxidized); (4) a hard and heavily mineralized quartz, containing pyrite and marcasite, with less galena, sphalerite, rhodochrosite, and rhodonite. It proved refractory; the Ag was present chiefly as argentite and stephanite; (5) ores like (4) except as regards the quantity of sulfides. It was most refractory of all and yielded only 50% of its Ag. The process formerly in use for unoxidized ore was chlorination with pan amalgamation, whereas the process adapted by the author is as follows: (1) crushing in soln.; (2) ordinary CN treatment; (3) chlorinating the residues with pan amalgamation. For the less refractory unoxidized ores, partial conc., fine grinding with 24 hrs. agitation and a thoro removal of soln. Results with the vacuum filter were very satisfactory. R. K.

The Present Status of the Basic Bessemer Process in Germany. W. ESSER. *Iron*Age, 86, 108-10; see C. A., 5, 1896.

L. A. TOUZALIN.

The Rationale of Dried Blast. J. W. RICHARDS. Iron Age, 86, 156-7; see C. A., 5, 1390.

L. A. TOUZALIN.

German Open Hearth Processes. R. Genzmer. Iron Age, 86, 224-6.—Abstract (see C. A., 5, 1896).

L. A. TOUZALIN.

The Best "All Around" Aluminium Mixture. Anon. Brass World, 7, 290.— An alloy containing 92% Al and 8% Cu gives the best results for general uses where an Al alloy is desired. It casts well, does not crystallize in service nor crack in the mold, and is sufficiently strong for most purposes. The Cu is best introduced by preparing a rich alloy of Al and Cu and melting together with the pure Al. Zn-Al alloys have been superseded on account of their tendency to form invisible cracks in the mold.

ROBERT KANN.

Rust. E. Donath and A. Indra. Chem. Ztg., 35, 773.—The % chem. comp. of 4 specimens of rust was found to be resp. as follows: Moisture, 4.41, 1.74, 2.35, 1.60; combined H<sub>2</sub>O, 12.21, 7.53, 6.23, 1.55; CO<sub>2</sub>, 0.83, 0.25, 0.65, 0.93; SiO<sub>2</sub>, 0.49, 0.46, 3.32, 0.84; P<sub>2</sub>O<sub>3</sub>, 0.46, 0.08, 0.43, 0.23; SO<sub>3</sub>, 2.72, 0.06, 0.18, 0.07; NH<sub>2</sub>, 1.07, 0.02, 0.06, 0.02; FeO, 2.23, 2.79, 6.04, 1.03; Fe<sub>2</sub>O<sub>3</sub>, 74.52, 86.45, 78.56, 92.94; Mn<sub>2</sub>O<sub>3</sub>, 0.23, 0.33, 1.69, 0.41; graphite, ?, ?, 0.49, ?. Sample No. 1 was from a steel bomb which had contained liquid SO<sub>2</sub> and had been exposed for a long time to at atm. in which SO<sub>2</sub> gas was being used. No. 2 was scale on the outside of a boiler

tube of malleable Fe which had been exposed to the atm. for a long time. No. 3 was from the base of a machine which had also been exposed to the atm. for a long time. No. 4 was from a strong wire cable. In No. 1, which contained considerable  $(NH_4)_2$ -SO<sub>4</sub> it was evident that the SO<sub>2</sub> in the atm. had a considerable influence upon the formation of the rust; this sample also contained 0.42% of FeSO<sub>4</sub>. Some sulfate was found in every sample of rust that the authors have examined. When the Fe is oxidized by rusting, the other constituents, S, P, Mn, etc., are also oxidized. NH<sub>2</sub> also seems to be present as NH<sub>4</sub> salt in all rust and each sample has contained some CO<sub>2</sub>. Altho it has been proved that CO<sub>2</sub> is not absolutely necessary for the formation of rust, still it is undoubtedly to be considered as an important factor. W. T. HALL.

Modifications which Nickel Steels Undergo on Long Heating or on Standing. CH.-ED. GUILLAUME. Compt. rend., 153, 156-60.—The change in vol. which Ni steels undergo upon long heating or upon long standing is illustrated by a curve and by a table which may serve as a guide for any one interested in the practical uses of these alloys. Prolonged heating at 100° places these alloys in a state of equil. irrespective of their previous treatment.

W. T. Hall.

The Marked Influence of Cepper in Iron and Steel on the Acid Corrosion Test.

W. H. WALKER. Mass. Inst. Tech. Eng. News, 66, 156.—In studying samples which had withstood corrosion for years but dissolved readily in acid, together with samples which were remarkably resistant to acid but corroded at a normal rate, it was found that the presence of Cu seemed to be the controlling factor in the resistance to soln. in acid. In other words, a slight tendency to dissolve in acid is not indicative of great purity.

W. T. HALL.

The Value of the Sulfuric Acid Corrosion Test. C. M. CHAPMAN. Eng. News, 66, 156.—Photographs are shown of 2 plates, one of improved Fe, and one of ordinary steel, showing results of weather exposure for 18 mos. Careful exam. after severe exposure failed to show any considerable difference in the corrosion and pitting that had taken place. The results of the  $H_2SO_4$  test on the same plates were as follows: At the end of the 1-hr. period prescribed by the test, the improved Fe had lost 0.003% while the common steel had lost 6.7%.

W. T. Hall.

Some Tests of the Rate of Corrosion of Metals Exposed in a Gaseous Atmosphere. A. W. CARPENTER. Eng. News, 66, 156.—To find a suitable metal for structural purposes and one which will resist corrosion when exposed to locomotive gases, W. Kittredge, of the N. Y. C. H. R. R. R., has made tests of the comparative rates of corrosion of various metals and especially of Fe and steel. Thin sheets of metal were exposed in a tunnel. In the first series of tests a plate of special Fe corroded at the rate of 1/2 in. per annum for each surface of 1/16 in. per plate with both surfaces exposed. In the second series of tests, resistance to corrosion was detd. by the following losses in wt. in g. per sq. in. of surface after 289 days' exposure: Special coated metal, o. 51; uncoated soft steel, 0.55; special galvanized Fe, 0.67; uncoated special Fe, 1.72. The amt. of this corrosion was less than that obtained in smokejack tests. In a third series of tests, the relative order of resistance for 58 days' exposure in a tunnel was as follows: (1) Special "A" Fe-sheets, plain, 0.047 av. loss in g. per sq. in.; (2) openhearth steel angles, plain, 0.060; (3) lead-coated, galvanized steel sheets, 0.067; (4) special "B" Fe sheets, plain, 0.077; (5) open-hearth steel bars, plain, 0.082; (6) wrought-Fe sheets, plain, 0.090; (7) open-hearth soft steel plates, plain, 0.095; (8) Bessemer steel angles, plain, 0.098; (9) special "A" Fe sheets, galvanized, 0.121; (10) special "B" Fe sheets, galvanized, 0.137; (11) Bessemer steel bars, plain, 0.147; (12) special non-ferric metal sheet, plain, 0.226.

The Strength of Steels in Compound Stress and Endurance under Repetition of

Stress. L. B. TURNER. Engineering, 92, 115-7, 183-5, 246-50. — The author has previously described (*Ibid.*, Feb.) some expts. carried out with the purpose of ascertaining how closely the elastic breakdown of a metal is dependent only upon the max. shear to which it is subjected. The present article is a sequel and embodies (1) some further tests with static pull and torque; (2) expts. on 3-dimensional stress; and (3) an investigation of the endurance of various steels under alternations of tension and shear.

W. T. Hall.

The Hardness of Quenched Steels. Portevin and Berjot. Rev. métal., Jan., 1910; Iron Age, 85, 726-7.—The Shore scleroscope enables one to det. the best reheating or tempering temp. for quenched steels. With case-hardened steels it gives the superficial hardness and does not appear to be influenced by the thickness of the case after the C has reached about 0.8%. The Brinell test depends on both the superficial hardness and the depth of case. A combination of both tests gives a good indication of the heat treatment and thickness of case. The max. hardness for case-hardened steels is obtained by quenching at 725° for ordinary and at 700° for Ni steel. The Ni steel used had about 2.5% Ni.

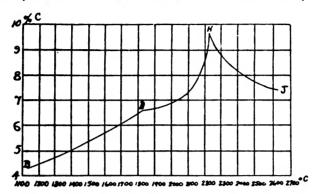
Some Recent Tests of Cast Iron. A. E. OUTERBRIDGE, Jr. Iron Age, 86, 56–7.— Two test bars  $1 \times 1 \times 15$  ins. and  $\frac{3}{4} \times \frac{3}{4} \times 15$  ins. were poured from the same ladle. Both bars were turned to a diam. of 0.505 in. between threaded ends and pulled in a 100,000 lbs. testing machine. Invariably the smaller bars ( $\frac{3}{4}$  in. sq.) of any series if free from flaws, showed higher tensil strength than the larger bars (1 in. sq.). An accompanying table shows that the min. increase in tensil strength of the small over the large bar is 2,250 lbs., and the max. 7,500 lbs. This sensitiveness of cast iron to cooling influences demonstrates the importance of adopting standard sizes of bars for testing purposes.

L. A. TOUZALIN.

Titanium and Segregation. G. B. WATERHOUSE. Iron Age, 86, 46-8.—Ti, when correctly applied, retards segregation of S, P and C in normally quick-setting steel. The third ingots of 2 sep. heats of Bessemer steel, one made with ferrotitanium and one without, were allowed to cool on their stools, and were then cut thru longitudinally. No trace of Ti could be found in the Ti treated metal. Diagrams of the analyses of both ingots show segregation as follows: In ordinary steel, ladle S, 0.098%; max. in ingot, 0.223%. In Ti steel, ladle S, 0.068%; max. in ingot, 0.101%. In ordinary steel, ladle P, 0.098%; max. in ingot, 0.167%. In Ti steel, ladle P, 0.093%; max. in ingot, 0.124%. In ordinary steel, ladle C, 0.44%; max. in ingot, 0.67%. In Ti steel, ladle C, 0.47%; max. in ingot, 0.69%. In all cases the area of segregation is greater in the ordinary steel. The 3 rails rolled from an ordinary ingot, and those from a Ti ingot, were drilled every 12-18 in, at side of head, to center. Analyses show that here too the use of ferrotitanium has lessened the segregation.

Copper Nickel Alloys. I. A. H. HIORNS. Metal. Ind., 9, 347-8.—This article deals with the influence exerted on the character of alloys by the degree of purity of their constituent metals. Com. Ni contains as chief impurities S, Fe, C and Si. The tendency for the C to assume the graphitic form is probably the main cause of the inability of such carburized Ni to assume a hardening form, like steel. Possibly a carbide of Ni exists, corresponding to the Fe<sub>2</sub>C, which has an embrittling effect. Another element which is said to have an embrittling effect on Ni is As. S should be absent from Ni which has to be pressed and rolled, and as com. Ni often contains S it is unsuitable for purposes where extreme ductility is required. Fe hardens, whitens, increases the strength, but reduces the elongation of Cu-Ni and German silver. It is very probable that C and Fe are the chief factors in rendering Ni brittle, either alone or by their influence on other impurities, thus confining its use to the production of German silver and Ni anodes for plating.

The Solubility of Carbon in Iron. O. RUFF AND O. GOECKE. Königl. Tech. Hochschule, Danzig. *Metallurgie*, 8, 417-21.—The authors have detd. the solubility of C in Fe at temps. between 1200 and 2600°. The Fe was heated in a graphite crucible (2.06% ash) in an evacuated elec. furnace until it was satd. with C, whereupon it



was quenched in ice water. The total C, graphite, and sp. gr. of the reguluses was detd. and the temp. observed optically by means of the Wanner pyrometer. The solubility curve BDHJ shows a max. at 2220°, corresponding to the comp. Fe<sub>2</sub>C, and a change in direction at 1887°, corresponding to the comp. Fe<sub>2</sub>C.

W. T. Hall.

The Resistance of Steels to Crushing and its Variations as a Function of Temperature. F. Robin. Rev. métal., 8, 423-35; cf. C. A., 5, 2390.—Tresca's law of static compression: "the specific work of equal impacts (kg. m. per cc.) produce equal crushing in solids of the same geometric form of the same material,"—has been applied to dynamic compression. The resistance to crushing is obtained by dividing the no. of kg. meters necessary to reduce the height of a cylinder 1/8, by the vol. of the test piece in cc. (original diam. of cylinder - height). The resistance curve is obtained by plotting the work performed by each shock against the total work necessary to produce equal deformation; the direction of the curve change; as ht. diminishes the resistance. Hard metals give hyperbolas which lie asymptotically to the axes of the coordinates; with soft metals such as Pb the curve turns toward the origin. In steels the curve for soft metal is obtained at 400°. The effect of crushing Fe-C alloys is discussed and curves are given for different steels containing varying % of Ni, Ni-Cr, Cr, W, and Mn. The presence of P increases resistance but variations are wider. A study of hammerhardness in relation to temp. showed a max. at 300°, a fall at 700°, and a variation from this point on depending on whether it is capable of taking temper. Forgeability, defined as diametric enlargement on crushing, when plotted against the temp., gives curves showing maximums at o° and 300° and minimum at 500°, then a rapid rise to 1000°, and finally a rapid fall. Ni, German silver and brass show a minimum at 300°. A Ni steel (30% Ni, 40% C, 1.0% Mn) gave an almost horizontal curve with a max. at 500°. E. J. WITZEMANN.

Fragility of Steels under Crushing: Micrographic Study of the Surfaces of the Bases of Cylinders Crushed at Different Temperatures. F. Robin. Rev. métal., 8, 436-45 (21 photomicrographs); see also preceding abstract.—(1) Fissures of intergranular fragility arise from the sepn. of grains of the metal. These do not extend across the grains unless the metal is subject to intragranular brittleness. This type of fissures is observed especially in coarse grain steels and P steels among others. (2) Fissures due to impurities or to loss of adhesion were found on the bases of some cylin-

ders. The nature of these fissures is difficult to det.: (1) they are terminated by rounded contours when the metal is very malleable, and have fine needle-point extremities when developed in the heart of fragil pieces; (2) they are generally radial or perpendicular to the specimen; (3) they do not usually follow the lines of deformation, but appear to originate in metal that has not been much disturbed. Two explanations of this second class of fissures are offered: (1) they are due to the enlargement of holes containing slags, oxides, or gases in the ferrite, for which considerable experimental evidence is offered; (2) they are due to loss of adhesion between bands of ferrite and are especially noticeable when the ferrite varies in constitution, as shown by unequal coloring with the reagents. C steels show fission in the pearlite; graphite of cast-iron is the origin of fissures.

E. J. WITZEMANN.

Assay of Silver-bearing Gouge Ores (KEYES, RIDDELL). 7. Determination of Vanadium in Steel and Iron (CRITES). 7.

BOUCHONNET, A.: Zink, Cadmium, Kupfer, Quecksilber. Paris: 402 pp., 5 Fr.

FENCHEL, A.: Metalikunde. Hamburg: Boysen & Maasch. 8°, 6.60 M.

HOOD, C.: Iron and Steel; their Production and Manufacture. New York: J. Pitman. 150 pp., 75 cents.

HORN, A.: Die autogene Schweiss und Schneidetechnik. Halle a/S, 8 M.

JONES, P. E.: Hardening and Tempering Steel. A workshop guide to the heat treatment of all steels. London: Cassell. 8°, 128 pp., 2 s.

KAISER, E. W.: Zusammenstelling der gebräuchlichen Metalliegierungen. Halle a/S, 2.80 M.

MORGAN, J. J.: Blast Furnace Practice. Lippincott. 53 pp., 75 cents.

Schott, E. A.: Neuere Sandstrahlgebläse für Gussputzertien. Berlin: H. Meusser. 8°, 48 pp., 2 M.

Wohlfahrts: Einrichtungen der Chemischen Fabrik und Zinnhütte. Essen-Ruhr: T. Goldschmidt.

Brit., 6,274, Mar. 12, 1910. J. W. BAILEY, E. 5th and Ingram Sts., Bayonne, N. J. App. for corroding lead, including a receptacle for the material, means for showering it across a body of air within the receptacle, and means for subjecting the material while suspended in or showering thru said body of air to repeatedly recurring violent impacts.

Brit., 9,227, June 21, 1910. B. J. SMART, Royal Arsenal, Woolwich. A low specific gravity alloy of Al, with a substantial % of Cu and Zn, contg. from about 0.1% to 2.0% of Mg and having high tensil strength and more or less ductility as required.

Brit., 11,503, May 9, 1910. W. H. FITZGERALD, 30 Franconia Road, Clapham S. W. A composition of materials for cooling excessively heated articles, such as gun barrels, consisting of a mixt. of soot and black lead (graphite) with or without the addition of H<sub>2</sub>O or oil to moisten the same.

Brit., 13,488, June 3, 1910. J. S. ISLAND, Cor. Brock Ave and Forence St., Toronto, Canada. Extracting the precious metals from ores by taking a quantity of ore finely pulverized and enclosing it within a receptacle, then effecting a union of Cl and SO<sub>2</sub> in their gaseous forms by passing them over an area of camphor and discharging the sulfuryl chloride so obtained into said receptacle and coincidently directing a flow of steam into said receptacle, thus providing 2 vaporous fluids which percolate thruout

the ore and in contact unite and create HCl and H<sub>2</sub>SO<sub>4</sub> thruout the body of ore, said acids acting on the precious metals in said ore body and converting them into sol. salts, and finally withdrawing the said ore body into leaching tanks for further treatment.

Brit., 14,192, June 11, 1910. A. L. BROOKE, 103 High St., Mortlake. Manuf. of an alloy of Al, Ni, and Mg, by reducing an O compd. of Ni to the metallic state in 1 crucible, m. the Al under a layer of C and argol in another crucible, pouring the molten metals into a heated third crucible, casting into ingots, and m. the alloy of Al and Ni so formed with the desired quantity of Mg under a layer of C and argol.

Brit., 14,940, June 21, 1910. B. J. SMART, Royal Arsenal, Woolwich. A low specific gravity alloy of Al and a substantial % of Zn and Cu, in which the Zn and Cu together constitute between about 10-20% of the alloy, the Cu being less than about 5% with or without a small quantity of Mg and having high tensil strength and more or less ductility as required.

Brit., 30,222, Dec. 29, 1910. J. NAULTY, 2616 Martha St., Philadelphia, Penna. An alloy comprizing Cu and Cr in the proportions of 9 parts Cu and 1 part Cr, combined with Ni 25% and Zn 25%.

Brit., 4,159, Feb. 18, 1911. A. C. HYDB, 3 Queens Gardens, Ealing. Obtaining molybdenum in a state which is ductil and malleable when cold, by adding to pure Mo a fraction of 1% of Fe, Ni, or Mn, and thereafter treating the compounded metals, forming and heating them in the elec. furnace by means of the elec. current, at a temp. approximately the m. p. of the added metal.

## 10. ORGANIC CHEMISTRY.

### J. BISHOP TINGLE.

The History of Iodogorgoic Acid. M. Henze. Chem. Physiol. Lab., Zoöl Station, Naples. Z. physiol. Chem., 72, 505-6.—Polemic and claims of priority in disclosing the constitution of this acid.

G. M. Meyer.

Action of Alkalis on Chloraloses. M. HANRIOT AND A. KLING. Compt. rend., 152, 1398–9.—In anhydrous media, Cl can be removed from chloraloses without entirely destroying the mol. Thus p-chloralose, heated 4–5 hrs. in sealed tubes with 4 parts MeOH satd. with NH<sub>3</sub>, gave 20% of a compd. C<sub>7</sub>H<sub>11</sub>O<sub>6</sub>CHCl<sub>2</sub>, dichloro-p-chloralose, needles, m. 156–7°, α<sub>D</sub> 10.57°; 7.73 g. dissolves in 100 g. 95% alc., 3.866 g. in 100 g. H<sub>2</sub>O at 18°. Dil. HCl hydrolyzes it to glucose and CHCl<sub>2</sub>CHO, while HNO<sub>3</sub> (d. 1.2) gives an amide, C<sub>7</sub>H<sub>9</sub>O<sub>5</sub>Cl<sub>2</sub>CONH<sub>2</sub>, pearly scales, m. 161–2°, which, on treatment in H<sub>2</sub>O with HNO<sub>2</sub>, evolves N and yields needles m. 129–30°, apparently consisting of a mixt. of the corresponding acid and lactone. The dichloro compd. yields, by the Baumann method, a tribenzoyl derivative, needles, m. 192°.

C. A. ROUILLER.

Action of Methylene Chloride on p,p-Ditolylmethane. James Lavaux. Compt. rend., 152, 1400-2.—Very pure p,p-ditolylmethane, m. 28°, obtained by a modification of Weiler's method (Ber., 7, 1181), consisting in the use of trihydroxymethylene instead of methylal, when condensed with 1.5 parts each of CH<sub>2</sub>Cl<sub>2</sub> and AlCl<sub>2</sub>, gave a mixture of 2,7- and 1,6-dimethylanthracenes, the latter predominating as in all reactions in which this mixt. is formed, showing that AlCl<sub>2</sub> produces a mol. transposition either in the ditolylmethane or the nascent dimethylanthracene and that the CH<sub>2</sub> groups in certain positions are more reactive towards AlCl<sub>2</sub>. Chas. A. ROUILLER.

Derivatives of Styrolene; Correction of some Experimental Errors. P. LEMOULT. Compt. rend., 152, 1402-4.—Having noted that the values given by Auwers, Roth

and Eisenlohr for the heats of combustion of styrolene derivs. (C. A., 4, 2143) did not agree with the values calc. by means of the author's formula (Ann. chim. phys., [8] 1, 469; 5, 5) for ethylene derivs., he has repeated some of their measurements and found that they were erroneous; the following are the values found by him: Styrolene, 1059.1;  $\beta$ -methylstyrolene, 1217.3;  $\alpha,\beta$ -dimethylstyrolene, 1373.4;  $\alpha,\beta$ -methylphenylstyrolene, 1939.0; diphenylstyrolene, 2510.3; di- $\beta$ -methoxystilbene, 2018.

CHAS. A. ROUILLER.

Catalytic Preparation in the Wet Way of Formenic Esters. J.-B. SENDERENS AND J. ABOULENC. Compt. rend., 152, 1671-3.—Equimol. amts. of AcOH and 95% EtOH b. alone 1 hr. gave 17.8% ester, while with 0.05 part anhydrous Al<sub>2</sub>(SO<sub>4</sub>)<sub>2</sub> 62.2% and with the hydrated salt 36% ester were obtained, and 0.01 and 0.05 part KHSO<sub>4</sub> yielded 64 and 82% ester, resp. H<sub>2</sub>SO<sub>4</sub>, whether 1, 2 or 10% by vol. was used, gave 86.5% ester, so that its influence cannot be explained as due to the heat liberated in the formation of hydrates but is catalytic and similar to that of Al<sub>2</sub>(SO<sub>4</sub>)<sub>2</sub> and KHSO<sub>4</sub>, EtHSO<sub>4</sub> (stable at the reaction temp.) being first formed and giving the unstable Et<sub>2</sub>SO<sub>4</sub> which, with the org. acid, forms the ester and regenerates EtHSO<sub>4</sub>.

CHAS. A. ROUILLER.

Action of Isobutylamine and of Diisobutylamine on α-Bromobutyric Acid. JEAN NIVIÈRE. Compt. rend., 152, 1673-4.—α-Isobutylaminobutyric acid is obtained by heating EtCHBrCO<sub>2</sub>H with 3 mols. of moderately dil. C<sub>4</sub>H<sub>6</sub>NH<sub>4</sub> in sealed tubes for 10 hrs. at 100-5° and distilling with 1 mol. Ba(OH)<sub>2</sub>; pearly lamellas, sublimes without m., loses CO<sub>2</sub> in HCl at 180-200°; hydrochloride, large crystals with 1.5 H<sub>2</sub>O, loses HCl at 100-10°; chloroplatinate, red needles with 2 H<sub>2</sub>O, decomp. 100-10°; chloroplatinate, golden yellow; copper salt, blue lamellas with 2 H<sub>2</sub>O, loses 1 H<sub>2</sub>O over H<sub>2</sub>SO<sub>4</sub>, becoming violet, decomp. 100-10°; silver salt, silky lamellas; picrate, yellow. Diisobutylamine and EtCHBrCO<sub>2</sub>H give only HOCHEtCO<sub>2</sub>H.

Reduction of Limonene. G. VAVON. Compt. rend., 152, 1675-7; cf. C. A., 4, 1478.—The vel. of absorbtion of H by limonene in the presence of Pt black and the change in the rotatory power of the liquid show that 2 ats. H are first absorbed to form a dihydride,  $C_{10}H_{18}$ , which then takes up 2 more ats., giving the compd.  $C_{10}H_{10}$ . The dihydride b. 175-7°,  $n_D^{18}$  1.4563,  $d_4^{18}$  0.8246,  $[\alpha]_{878}$  118°,  $[\alpha]_{498}$  234°. With Br it gives a dibromide,  $b_{16}$  136-40°,  $n_D^{21}$  1.5236,  $d_4^{21}$  1.459,  $[\alpha]_{878}$  49°,  $[\alpha]_{490}$  100°. Nitrosochloride,  $C_{10}H_{18}$ NOCl, m. 95-6°,  $[\alpha]_{878}$  344°,  $[\alpha]_{496}$  724°. Chas. A. Rouiller.

Reduction of Carvone. G. VAVON. Compt. rend., 153, 68-71; cf. preceding abstr.—The reduction of carvone proceeds in 3 steps, 2, 4 and 6 ats. H being successively absorbed, and by stopping the reaction at the proper instant it is easy to obtain the intermediate products: Carvotanacetone, b. 227-8°,  $d_1^{18}$  0.937,  $n_D^{18}$  1.4817,  $[\alpha]_{h78}$  59.8°,  $[\alpha]_{436}$  141°,  $R_m$  46.20. Tetrahydrocarvone, b. 218-9°,  $d_1^{20}$  0.904,  $n_D^{20}$  1.4555,  $R_m$  46.25,  $[\alpha]_{h78}$  —27°,  $[\alpha]_{436}$  —70.6°. Oxime, m. 100°,  $[\alpha]_{h78}$  —40.5°,  $[\alpha]_{436}$  —82.5°. Carvomenthol, b. 217-8°,  $d_1^{20}$  0.908,  $n_D^{20}$  1.4648,  $R_m$  47.49,  $[\alpha]_{h78}$  —24.7°,  $[\alpha]_{436}$  —47.4°. Acetate, b. 230-1°,  $d_1^{20}$  0.928,  $n_D^{20}$  1.4477,  $R_m$  57.07,  $[\alpha]_{h78}$  —27.6°,  $[\alpha]_{436}$  —52.4°. Benzoate,  $b_{15}$  185-6°,  $d_1^{20}$  1.006,  $n_D^{20}$  1.509,  $R_m$  77.19,  $[\alpha]_{h78}$  —12.9°,  $[\alpha]_{436}$  —22.8°. Quite a number of other ketones and aldehydes have been reduced in this way.

Azomethins Derived from Phenylisoxazolone. ANDRA MEYER. Compt. rend., 152, 1677-80; cf. C. A., 2, 1718.—When phenylisoxazolone is heated in alc. with an equimol. amt. of a p-nitrosoamine, the mixt. changes to a dark violet and dyes of the type (I) are pptd. almost quant. These are insol. in H<sub>2</sub>O, only slightly in cold alc.; decompd. by alk.; sol. with purplish violet color in conc. H<sub>2</sub>SO<sub>4</sub>. Diethylaminophenyliminoketophenylisoxazolone, steel-gray prismatic needles, m. 117° (decomp.). Methylethylaminophenyliminoketophenylisoxazolone, purplish black needles with bluish reflec-

tion, m. 143° (decomp.). Phenylaminophenyliminoketophenylisoxazolone, violetblack needles with greenish reflection, m. 141-2° (decomp.). With nitrosopyrazoles

instead of nitrosamines are obtained compds. of the type (II), insol. in H<sub>2</sub>O, sol. in org. solvents with intense violet-red color, which (in dil. AcOH or alc.) soon disappears owing to hydrolysis. 3,5-Dimethyl-4-pyrazoleiminoketophenylisozazolone, vermillion-red needles, m. 140° (decomp.), sol. in alc. alkali or NH<sub>2</sub> with violet, in conc. H<sub>2</sub>SO<sub>4</sub> with dark red color. 1-Phenyl-3,5-dimethyl-4-pyrazoleiminoketophenylisozazolone, light orange-brown needles, m. 157° (decomp.), forms ruby-red solns. 3-Methyl-5-phenyl-4-pyrazoleiminoketophenylisozazolone, light red needles, m. 99° (decomp.), 3-Methyl-1,5-diphenyl-4-pyrazoleiminoketophenylisozazolone, dark red-brown needles, m. 143-4°. With nitrosoantipyrine is obtained antipyryliminoketophenylisozazolone (III), scarlet-red prismatic lamellas, m. 147-8° (decomp.), sol. in conc. H<sub>2</sub>SO<sub>4</sub> with

orange-yellow color. Tolypyryliminoketophenylisoxazolone, C<sub>n</sub>H<sub>18</sub>O<sub>2</sub>N<sub>4</sub>, m. 152° (decomp.). Ureinoindamine (IV), from alloxan and aminoantipyrine, dark violet microcryst. powder, decomp. about 300°, sol. in H<sub>2</sub>SO<sub>4</sub> with yellow color. C. A. R.

Dehydration of Alkyl- and Benzylpseudobutylphenylcarbinols. PAULINE LUCAS. Compt. rend., 152, 1771-4; cf. C. A., 4, 2095.—The hydrocarbon resulting from the dehydration of methylpseudobutylphenylcarbinol is oxidized by CrO<sub>3</sub> to AcPh and Me<sub>3</sub>CBz, together with a little MeAc, and must therefore consist of a mixture of Me<sub>3</sub>-CPh: CH<sub>3</sub> and Me<sub>3</sub>C—CPhMe. Ethylpseudobutylphenylcarbinol, HOCPhEtCMe<sub>3</sub>,

b<sub>18</sub> 115-6°, gives an apparently homogeneous dehydration product, Me<sub>3</sub>CCPh: CHMe, b<sub>12</sub> 90-5°, yielding CO<sub>2</sub> and Me<sub>3</sub>CBz on oxidation. The hydrocarbon from pseudo-butylbenzylphenylcarbinol is Me<sub>3</sub>CCPh: CHPh, yielding, on oxidation, CO<sub>2</sub>, BzOH, Me<sub>3</sub>CBz and a compound C<sub>18</sub>H<sub>20</sub>O, m. 131°, b<sub>18</sub> 195-200°, which forms neither an oxime nor a semicarbazone.

CHAS. A. ROUILLER.

α-Glucodecose and α-Glucodecite. L.-H. PHILIPPE. Compt. rend., 152, 1774-6; cf. C. A., 5, 1263.—Glucodecose, obtained by treatment of glucodeconic acid with 2.5% Na-Hg in acid soln. at  $-2^{\circ}$ , needles, m. about 210°; 0.5 g. dissolves in 100 g. boiling 85% EtOH, 22 g. in 100 g. H<sub>2</sub>O at 20°;  $[\alpha]_D^{20}$  37° (fresh 10% soln.), 50.4° after 24 hrs. or on b. It sometimes crystallizes in hexagonal lamellas with 1 H<sub>2</sub>O. Its reducing power is about 76% of that of glucose. Phenylhydrazone, prismatic needles, m. about 278°. α-Glucodecite, from the decose and Na-Hg first in acid, then in neutral, soln., prismatic needles, m. 222° (it is appreciably volatil at this temp.); 0.4 g. dissolves at ordinary temp. and 20 g. at 100° in 100 g. H<sub>2</sub>O;  $[\alpha]_D^{18}$  1.2°. Acetyl derivative,  $C_{10}H_{19}O_{10}Ac_{10}$ , rectangular lamellas, m. 149-50°,  $[\alpha]_D^{18}$  16.0° (5% CHCl<sub>3</sub> soln.).

Ketones of the Benzyldimethylacetophenone Type. Trialkylacetic Acids and Trialkylethyl Alcohols Formed from Them. A. HALLER AND ÉDOUARD BAUER. Compt. rend., 153, 21-7; cf. C. A., 3, 1161, 2676.—o-Xylyldimethylacetophenone, MeC<sub>c</sub>H<sub>4</sub>CH<sub>2</sub>-CMe<sub>2</sub>Bz, b<sub>1s</sub> 199-200°, obtained in 88-90% yield by b. MeC<sub>c</sub>H<sub>4</sub>CH<sub>2</sub>Br for 2 hrs. with

Me, CHBz previously treated with NaNH, in Et.O. The o-xylyl bromide b, 108°, and is obtained practically free from dibromide by treating o-xylene vapors with  $\frac{3}{2}$  mol. Br. m-Xylyl bromide b<sub>12</sub> 105°. m-Xylyldimethylacetophenone, b<sub>12</sub> 196-7°; yield, 75%. p-Xylyl bromide, m. 35°, b<sub>10</sub> 100°. p-Xylyldimethylacetophenone, b<sub>12</sub> 200-2°; yield, 80%. p-Methoxybenzyldimethylacetophenone, b<sub>18</sub> 222-4°, obtained in 60% yield from BzCMe<sub>2</sub>Na and anisyl chloride. Heated in dry C<sub>2</sub>H<sub>4</sub> with NaNH<sub>2</sub> 4-5 hrs., the above ketones give the following amides: o-Xylyldimethylacetamide, pearly scales, m. 62-3°, b. 188-92°. m-Xylyldimethylacetamide, needles, m. 46-7°. p-Xylyldimethylacetamide, needles, m. 85-6°. p-Methoxybenzyldimethylacetamide, needles from Et.O, m. 72°, solidify and m. again 99-100°, b<sub>18</sub> 218-25°. When recrystallized, it again m. 72°, but after standing over H2SO4 it m. 99-100°; it therefore probably sep. with Et.O. o-Xylyldimethylacetic acid, obtained by Bouveault's method, m. 48°, b, 180-1°. m-Xylyldimethylacetic acid, b<sub>16</sub> 178°. p-Xylyldimethylacetic acid, m. 53-4°, b<sub>16</sub> 180°. p-Methoxybenzyldimethylacetic acid, m. 52-3°. The above amides, when treated with Na and abs. alc., give about 80% of the corresponding alc. and 5-7% of amine. o-Xylyldimethylethyl alcohol, b<sub>18</sub> 142-3°. o-Xylyldimethylethylamine, b<sub>18</sub> 129-30°, rapidly absorbs CO2; chloroplatinate, dark yellow crystals. m-Xylyldimethylethyl alcohol, b14 139-40°. m-Xylyldimethylethylamine, b<sub>11</sub> 134-5°. p-Xylyldimethylethyl alcohol, needles, m. 37°, b, 141°. p-Methoxybenzyldimethylethyl alcohol, m. 50°.

Hydroxythiophenes. MAURICE LANFRY. Compt. rend., 153, 73-6.—Thiophene (1-3% AcOH soln.) and com. H<sub>2</sub>O<sub>2</sub> (6-13 vol. % O) were b. in various proportions under a reflux condenser. When more than 1.5 g. active O per g. of thiophene is used, a 30% yield is obtained of a mixt. of dihydroxythiophene, b. about 130°, do 1.26, and tetrahydroxythiophene, b. 158-60°, d<sub>20</sub> 1.43. Both are insol. in H<sub>2</sub>O, very sol. in org. solvents, unchanged by b. alk. or by HNO, up to 100°, but boiling HNO, (36° Bé.) or cold furning acid violently oxidize them to H<sub>2</sub>SO<sub>4</sub>. H<sub>2</sub>SO<sub>4</sub> (66° Bé.) and acid containing 10% SO, react similarly. PhNHNH, does not react with them; with isatin in H<sub>2</sub>SO<sub>4</sub> a green color is formed, changing to blue on the addition of a little H<sub>2</sub>O and disappearing as more is added. Conc. H.O. oxidizes them to H.SO.; Zn or Na in AcOH reduces the dihydroxy compd. to thiophene. With Br the tetrahydroxy deriv. gives tetrabromotetrahydroxythiophene tetrabromide, C4O4Br4S, needles, m. 65-6°. If less than 1.5 g. active O per g. of thiophene is used, a puce-colored ppt. is soon formed whose comp. varies with the length of the reaction, becoming poorer in S as the oxidation continues (after 15 min. it has approx. the comp. (C,H2OS), ; insol. in practically all solvents, partially sol. in AcMe, sol. in alk. and NH, and repptd. by acids (but not by CO<sub>2</sub>) in brown flocks; insol. in H<sub>2</sub>SO<sub>4</sub> (66° Bé.). Conc. H<sub>2</sub>O<sub>2</sub> (100 vols. O) has no action on thiophene. CHAS. A. ROUILLER.

Catalytic Oxidation of Phenols in the Presence of Iron Salts. H. Colin and A. Sénéchal. Compt. rend., 153, 76–9.—The progress of oxidation in solns. containing 0.5 mol. of hydroquinone, 0.85 mol.  $H_2O_3$ , 0.0001 mol. FeCl<sub>3</sub> and 0.004–4.0 mols.  $H_2SO_4$  was observed by colorimetric comparison with a standard soln. As the conc. of the  $H_2SO_4$  increases, the velocity of oxidation decreases to a minimum and then increases, probably owing to the intermediate formation of Caro's acid. When the conc. of the acid reaches 0.08 N crystals of quinhydrone begin to sep., increasing for a time and then beginning to decrease as the conc. of acid increases. Beyond 0.2 N conc. of acid the oxidation is so much retarded that quinhydrone is practically the sole product of oxidation and the results for the velocity of the reaction obtained by weighing the amt. of the quinhydrone agree with the values obtained by the colorimetric method. Qual. results of a similar character are obtained with HCl or HNO<sub>3</sub> instead of  $H_2SO_4$ , and with  $H_2SO_4$ , instead of  $H_2SO_4$ . With AcOH the retarding influence becomes noticeable only at high conc., no quinhydrone being formed. Beyond conc. of 14.0 N

the peroxidase system FeCl<sub>2</sub> + H<sub>2</sub>O<sub>2</sub> is completely paralyzed. (CO<sub>2</sub>H)<sub>2</sub> has a much greater influence, about 40 times as great as that of H<sub>2</sub>SO<sub>4</sub> for a conc. of 0.0076 N. Citric acid is intermediate between AcOH and (CO<sub>2</sub>H)<sub>2</sub>. These results show that the retarding influence of the acids is due to their action on the Fe salt, forming complex salts in which the oxidizing properties of the Fe are not manifested and which, in the case of the strong acids (ferrisulfates), are stable only in the presence of excess of acid, while in the case of weak acids (ferriacetates, ferrioxalates, ferricitrates) they resist hydrolysis, even in dil. soln.

Chas. A. Rouiller.

Lactonization of  $\alpha$ -Ketonic Esters. H. GAULT. Compt. rend., 153, 107-10.— Ethyl- $\alpha$ -keto- $\gamma$ -valerolactone- $\gamma$ -carboxylate, slightly yellow liquid, is easily obtained by the action of NaOEt, amines, or H<sub>2</sub>SO<sub>4</sub> on Et pyruvate. Phenylhydrazone, m. 120-1°. Semicarbazone, m. about 220°. Ethyl oxalylcitric lactone is obtained from Et<sub>2</sub>NH, Et oxalacetate amd NaOEt. Ethyl  $\alpha$ -keto- $\gamma$ -valerolactone- $\beta$ -acetic- $\gamma$ -propionic- $\gamma$ -carboxylate, liquid which cannot be distilled, is obtained by either of the three methods given above. It seems to react only in the enolic form, giving a non-cryst. benzoyl derivative, and is very easily saponified by boiling dil. HCl with loss of CO<sub>2</sub>. Ethyl  $\alpha$ -keto- $\gamma$ -valerolactone- $\beta$ -propionic- $\gamma$ -butyric- $\gamma$ -carboxylate has similar properties. C. A. R.

Preparation of Unsymmetrical Benzyldialkylacetic Acids. Ph. Dumesnil. Compt. rend., 153, 111-3.—Methylethylacetophenone, obtained by b. EtBz in 5 vols. C<sub>6</sub>H<sub>6</sub> for 5 hrs. with 1 mol. NaNH<sub>2</sub> and then 1 hr. with 1 mol. EtI, b<sub>10</sub> 109°. Methylethylbenzylacetophenone, b<sub>16</sub> 201°; boiled 6 hrs. in Me<sub>2</sub>C<sub>6</sub>H<sub>4</sub> with 1 mol. NaNH<sub>2</sub> and a trace of H<sub>2</sub>O, it gives methylethylbenzylacetamide, yellow liquid, hydrolyzed by heating 10 hrs. at 150° with 50% H<sub>2</sub>SO<sub>4</sub> to methylethylbenzylacetic acid, tables, m. 31°, b<sub>16</sub> 180°. Ethylpropylacetophenone, b<sub>16</sub> 138°. Ethylpropylbenzylacetophenone, prisms, m. 52°, b<sub>20</sub> 223°; with NaNH<sub>2</sub> and a trace of H<sub>2</sub>O, it gives a little amide, but the chief product is 3-benzylhexane, b<sub>16</sub> 117°. Ethylpropylbenzylacetic acid, b<sub>16</sub> 200° C. A. R.

Constitution of some Nitro Derivatives Obtained by the Action of Nitric Acid on Aloins. E. LAGER. Compt. rend., 153, 114-6; cf. C. A., 5, 1400.—Pure chrysammic acid gives m-hydroxytrinitrobenzoic acid with HNO, and in greater amt. than tetranitro-aloemodin, so that in the formation of the nitrobenzoic acid from the latter chrysammic acid must be an intermediate product, a fact which can be explained by Robinson and Simons' formula (J. Chem. Soc., 25, 76) for this acid. The correctness of this being established, the formula of aloemodin can be deduced and makes it probable that in barbaloin the d-arabinose group is attached to the 1-OH group in the aloemodin and in isobarbaloin to the 8-OH group, the alcoholic OH group, in both cases, remaining unaffected.

CHAS. A. ROUILLER.

Dimethylpentane Obtained by Heating a Dimethylcaoutchouc. A.-H. RICHARD. Compt. rend., 153, 116-20.—2,3-Dimethyl-1,3-butadiene (methylisoprene, Couturier's hydrocarbon) behaves in its polymerization like isoprene, giving under the influence of light or heat, a caoutchouc-like mass, which, on dry distillation, yields chiefly a homoterpene,  $C_{12}H_{20}$ ,  $b_{22}$  97-8°, b. about 205°, do 0.872,  $d_4^{25}$  0.85322,  $n^{\alpha}$  1.47463,  $n_{D}$  1.47786,  $n_{B}$  1.48572,  $n_{T}$  1.49241, optically inactive, easily polymerized, gives an intense red color with AcOH-H<sub>2</sub>SO<sub>4</sub>. Nitrosochloride hydrochloride,  $C_{12}H_{20}$ ONCl.HCl, yellow powder, decomp. about 160°. In the presence of Pt black the homoterpene absorbs 2 ats. of H, giving the compound  $C_{12}H_{22}$ ,  $b_{20}$  93-5°, do 0.860,  $d_4^{25}$  0.842307,  $n_{\alpha}$  1.46363,  $n_{D}$  1.47299,  $n_{T}$  1.47856, The compds.  $C_{12}H_{20}$  and  $C_{12}H_{22}$  therefore prob-

ably have the formulas (I) and (II), resp.

CHAS. A. ROUILLER.

Action of the Light of the Mercury Lamp on Chlorophyll Solutions. H. BIERRY AND J. LARGUIER DES BANCELS. Compt. rend., 153, 124-5.—EtOH-H<sub>2</sub>O solns. of chlorophyll from fresh spinach leaves placed between 2 Hg lamps in such a way that the temp. did not exceed 45°, became yellowish after 24-48 hrs. and no longer showed the absorption bands of chlorophyll. After 3-5 days Grimbert's test (C. A., 5, 1794) showed small amts. of urobilinogen.

Syntheses of Substituted  $\beta$ -Diketones, Ketonic Esters and Enolic Esters by means of Sodiated Ketones. A. HALLER AND ED. BAUER. Compt. rend., 153, 145-52.-BzCNaMe, and BzCl give 2 products: (1) Dibenzoyldimethylmethane, m. 99-100°, b<sub>14</sub> 195-6°, decompd. by alc. KOH into BzOH and BzCHMe<sub>2</sub>; oxime, BzCMe<sub>2</sub>CPh: NOH, m. 166°; and (2) 1-phenyl-2-methyl-1-propene-1-ol benzoate, BzOCPh: CMe, needles, m. 53-4°, b<sub>12.4</sub> 194-6°, decomp. by 1% HCl in MeOH into BzOMe and BzCH-Me<sub>a</sub>. Benzoylpivaloyldimethylmethane, m. 35°, b<sub>16</sub> 158°; oxime, probably HON: CPhCMe<sub>2</sub>COCMe<sub>2</sub>, m. 178°. Together with the ketone are formed small amts. of its isomer, 2,4,4-trimethyl-2-pentene-3-ol benzoate, Me<sub>2</sub>CC(OBz): CMe<sub>2</sub>. Pivaloyl chloride, Me<sub>2</sub>CCOCHMe<sub>2</sub> and NaNH<sub>3</sub>, in C<sub>6</sub>H<sub>6</sub>, give a compd. C<sub>12</sub>H<sub>24</sub>O<sub>3</sub>, b<sub>16</sub> 102°, b. 213-4°, which, while it does not give an oxime or semicarbazone, doubtless consists of octomethylacetylacetone and its enolic isomer. CH<sub>2</sub>ICH<sub>2</sub>CO<sub>2</sub>Et and BzCNaMe, give 55-60% of ethyl 7-benzoyl-7-methylvalerate, b<sub>13</sub> 183°; oxime, needles, m. 119-20°. 7-Benzoyl-7-methylvaleric acid, oil; silver salt, white ppt.; oxime, m. 123-4°. Ethyl 7-benzoylcaproate, bis 189-91°; p-nitrophenylhydrazone, dark orange crystals, m. about 205°. 7-Benzoylcaproic acid, oil which cannot be distilled; silver salt, white ppt., CH. ICH. CO.H and Me, CCOCNaMe, react energetically in C.H., but no definit product could be isolated.

New Preparations of Benzylamines and Hexahydrobenzylamine. PAUL SABATHER AND A. MAILHE. Compt. rend., 153, 160-2; cf. C. A., 3, 2145.—In the presence of NH<sub>2</sub> and ThO<sub>2</sub>, PhCH<sub>2</sub>OH vapors are converted chiefly into mono-, di- and tribenzylamines, the mono compd. predominating if the reaction is carried out at 330° and the dibenzyl compd. if at 370-80°. The gradual deposition of the resinous hydrocarbon  $(C_7H_6)_z$  on the ThO<sub>2</sub> (C. A., 2, 3058) makes it necessary to renew the catalyzer occasionally by ignition at a dark red heat. The PhCh<sub>2</sub>NH<sub>2</sub> so obtained, when reduced over Ni at 170-80°, gives, besides some NH<sub>2</sub> and PhMe, considerable hexabenzylamine, b. 162°, intensely alkaline, giving a carbonate, rhombohedral laminas; hydrochloride, lamellas. With PhNCO it easily forms phenylbenzylurea, m. 152°. C. A. R.

Isopyromucic Acid. Action of Oxidizing Agents. Dibromomaleic and Bromo-hydroxymaleic Dialdehydes. G. Chavanne. Compt. rend., 153, 185-8; cf. Ann. chim. phys., 3 (1904).—Dil. alk. H<sub>2</sub>O<sub>2</sub> decomp. isopyromucic acid into maleic acid and CO<sub>2</sub>, together with a little HCO<sub>2</sub>H. Bromoisopyromucic acid, C<sub>2</sub>H<sub>2</sub>O<sub>3</sub>Br, dissolves in Br, forming a compd. C<sub>5</sub>H<sub>2</sub>O<sub>3</sub>Br<sub>2</sub>, sepg. in an unstable form which is spontaneously transformed into stable, short, yellow prisms, m. 88-9° and decomp. into Br and the original acid slightly above the m. p. With cold H<sub>2</sub>O it gives HBr and a comp. C<sub>5</sub>H<sub>4</sub>O<sub>4</sub>Br<sub>2</sub>, decomp. 175°. The comp. C<sub>5</sub>H<sub>2</sub>O<sub>3</sub>Br<sub>3</sub>, when dissolved in Br and poured into a little H<sub>2</sub>O, loses CO<sub>2</sub> and forms dibromomaleic dialdehyde, OHCCBr:CBrCHO, m. 34°, which, when heated with 12-15 parts of H<sub>2</sub>O, gives bromohydroxymaleic dialdehyde, OHCC(OH):CBrCHO, scales, m. 83-3.5°; acetate, b<sub>1.6</sub> 118-20°; semicarbazone, decomp. 198°; phenylhydrazone, prisms (probably orthorhombic), decomp. 126-6.5°; trioxime, m. 94-5°.

Transformation of Substituted Paraconic Acids into Isomeric Cyclopropanedicarboxylic Acids. Ph. Barbier and R. Locquin. Compt. rend., 153, 188-91.—Phenylparaconic acid, heated 12 hrs., in 1.5 parts C<sub>6</sub>H<sub>6</sub>, on the H<sub>2</sub>O bath with SOCl<sub>2</sub>, is transformed into phenylcyclopropanedicarboxylic anhydride, whose anitide (I) m. 178-

80° and anil (II) m. 191-3°. Dimethylparaconic acid gives dimethylcyclopropanedi-

carboxylic anhydride, whose anilide m. 153° and anil, needles, m. 115°. The reaction is explained as taking place through the intermediate compds. RR'C(OSOCI)CH-(CH<sub>2</sub>COCI)CO<sub>2</sub>H and RR'C — CHCO<sub>2</sub>H.

HCOCI CHAS. A. ROUILLER.

Cellulose Nitrites. M. MARQUEYROL AND D. FLORENTIN. Bull. soc. chim., 9, 306-9.—Cellulose (in the form of viscose or Cu silk or absorbent cotton), allowed to stand a long time in contact with HNO<sub>2</sub> (either dil. or conc.) containing more than enough urea nitrate to destroy any nitrous vapors which might have been present in the acid or have been formed in the reaction, gives nitrate esters low in N, practically insol. in MeAc and yielding abundant amts. of nitrites on sapon. with KOH. These and other known facts make the work of Nicolardot and Chertier (C. A., 5, 1588) seem erroneous.

Mixed Organo-metallic Derivatives of Zinc and their Use in Organic Synthesis. E. E. Blaise. Bull. soc. chim., 9, I-XXVI (May 5).—Address delivered before the Soc. chim. France, Mar. 18, 1911, giving an account of the author's work. C. A. R.

Preparation of Mesoxalic Esters. André Meyer. Coll. France, lab. chim. org. Bull. soc. chim., 9, 423-5.—The low yields often obtained by the Schmitt (Compt. rend., 140, 1400) and Curtiss (C. A., 2, 2948) methods for the prep. of mesoxalic esters are due to the formation of an unstable intermediate compd.; when, in an operation in which the yield is poor, the residues are heated at ordinary pressure, there is a copious evolution of nitrous fumes and a further amt. of the mesoxalic ester is obtained. By using carefully dried N<sub>2</sub>O<sub>2</sub> (preferably from 400 g. NaNO<sub>2</sub> and 700 g. H<sub>2</sub>SO<sub>4</sub> for 150 g. malonic ester) and allowing the bluish green liquid to stand several days at ordinary temp., protected from moisture, the yield of mesoxalic ester is generally 85-90%, the slightest trace of moisture leading to the formation of considerable amts. of nitromalonic esters and various oxidation products.

Chas. A. Rouiller.

Acyclic Diacid Ketones. I, II. E. E. BLAISE AND H. GAULT. Bull. soc. chim., 9, 451-8, 458-64.—Triethyl oxalsuccinate, b<sub>13</sub>-13 170-5°, heated 8 hrs., in the form of its Na salt, on the H<sub>2</sub>O bath with 1.05 mols. PrI, gives a 25-30% yield of triethyl O-propyloxalsuccinate, EtO<sub>2</sub>CC(OPr): C(CO<sub>2</sub>Et)CH<sub>2</sub>CO<sub>2</sub>Et, b<sub>13</sub> 190-1°; dil. acids hydrolyze it to α-ketoglutaric acid, which m. 112-3° and is colored greenish yellow in alc. by FeCl<sub>3</sub>; phenylhydrazone, slightly yellow crystals, m. about 260° (decomp.); semicarbazone, m. about 220°; pyridazinonecarbaxylic acid (I), prisms, m. 197° (decomp.).

Diethyl ketoglutarate, b<sub>18</sub> 144°; semicarbazone, m. 114°. Triethyl oxalpyrotartrate, viscous liquid which cannot be distilled or titrated directly with alk., colors FeCl<sub>2</sub> dark red; it is hydrolyzed by dil. acids to α-methyl-α'-ketoglutaric acid, viscous liquid; oxime, m. about 162° (decomp.), extremely unstable; semicarbazone, m. 141-2° when heated slowly, 156° when heated rapidly; p-nitrophenylhydrazone, m. 163°; phenyl-kydrazone, S-yellow crystals, m. 171-2°, loses H<sub>2</sub>O at 140°, giving 1-phenyl-5-methyl-6-pyridasinone-3-carboxylic acid (II), lamellas, m. 134°; while with N<sub>2</sub>H<sub>4</sub>-H<sub>2</sub>O the keto

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acid gives directly 5-methyl-6-pyridazinone-3-carboxylic acid, m. 175° (decomp.); if heated slowly, m. about 192° (decomp.). Diethyl  $\alpha$ -methyl- $\alpha'$ -ketoglutarate, b<sub>18</sub> 144-5°; bisulfite addition product, C<sub>10</sub>H<sub>18</sub>O<sub>8</sub>.HNaSO<sub>2</sub>.12H<sub>2</sub>O, micaceous needles; semicarbazone, m. 98°; p-nitrophenylhydrazone, m. 109-10°. In the prep. of the oxalpyrotartrate, a small amt. of propane- $\alpha,\beta,\beta$ -tricarboxylic acid, m. 170° (decomp.) is obtained in the form of the tri-Et ester; on heating it gives pyrotartaric acid.

CHAS. A. ROUILLER

Diacid Ketones. III. E. E. BLAISE AND GAULT. Bull. soc. chim., 9, 588-92. —[EtO<sub>2</sub>CCOCH(CO<sub>2</sub>Et)], is easily hydrolyzed by conc. HCl, giving a 65% yield of  $\beta$ -hydroxy- $\alpha$ -pyrone- $\alpha'$ -carboxylic acid (I), colors FeCl, green and a pine splinter dipped in HCl violet, gives no ketone reactions but has a marked reducing power. Heated at 150°, it sublimes, losing CO<sub>2</sub> and forming isopyromucic acid (II), needles, m. 87°.

These facts show that dioxalsuccinic lactone is a  $\delta$ -lactone (III) and not a  $\gamma$ -lactone as assumed by Wislicenus and Boekler (Ann., 245, 11). Chas. A. ROUILLER.

Ketoglutaric Acids and Aldehyde Acids of the Succinic Series. E.-E. BLAISE. Compt. rend., 153, 71-3; cf. preceding absts.—When Et oxalpyrotartrate is allowed to stand several wks. at the ordinary temp., with HBr satd. at 0°, the crude sapn. product can be sepd. by repeated distillations into 3 fractions: (1) Chiefly methylketoglutaric lactone, MeCH.CH: CCO<sub>2</sub>H, b<sub>11</sub> 82-4°; (2) Ethyl pyrotartrate aldehyde

EtO<sub>2</sub>CCHMeCH<sub>6</sub>CHO, b<sub>18</sub> 89-90°, colors Schiff's reagent at once; semicarbazone, m. 110.5°, p-nitrophenylhydrazone, m. 89°, oxime, b<sub>18</sub> 137-8°, pyridazinone, m. 66°, b<sub>18</sub> 134-6°; and (3) Pyrotartaricethoxylactone, b<sub>18</sub> 101°, possesses no aldehyde properties but on hydrolysis yields pyrotartaric acid aldehyde, viscous liquid, b<sub>12·3</sub> 139-40°, colors Schiff's reagent a reddish violet, reduces NH<sub>2</sub>-AgNO<sub>3</sub>, miscible with H<sub>2</sub>O, EtOH and Et<sub>2</sub>O; semicarbazone, m. 159°; oxime, m. 77°; p-nitrophenylhydrazone, m. 198°; phenylhydrazone, m. 71-2° and on distillation in vacuo yields the methylphenylpyridazinone, m. 42°, b<sub>12</sub> 183-5°. Chas. A. Rouiller.

Catalytic Reduction of Cyclic Oximes. Synthesis of Aryl Amines. A. MAILHE AND M. MURAT. Bull. soc. chim., 9, 464-8.—PhMeC: NOH vapors passed over Ni with H, at 250-70°, gave a little PhMeCHNH, about 20% of BzMe, and some di-α-phenylethylamine, (PhMeCH)2NH, d13 1.018, nD 1.573, RD 72.2, b. 295-8°. PhEtC: NOH gives 70% of a mixt. of PrPh and PhCH: CHMe, a little PhEtCHNH, and about 15% of di-α-phenylpropylamine, b. 310-5°; hydrochloride, m. about 200° (decomp.). PhPrC: NOH yields a little α-phenylbutylamine, b. 240°, d<sub>13</sub> 0.9813, n<sub>D</sub> 1.534, R<sub>D</sub> 47.8, and  $di-\alpha$ -phenylbutylamine, b. about 320°, together with considerable amts. of PhBu and PhCH: CHEt. From Ph.C: NOH are obtained more than 70% Ph. CHNH, and some dibenzhydrylamine, m. 126°; hydrochloride, m. about 300° (decomp.). o-Methylcyclohexanone oxime gives some o-methylcyclohexylamine, MeC<sub>6</sub>H<sub>10</sub>NH<sub>2</sub>, b. 150°, methylcyclohexanone and a little di-o-methylcyclohexylamine, b. about 260° (decomp.); hydrochloride, m. about 225° (decomp.). The m-compd. reacts similarly; di-m-methylcyclohexylamine, b. 265° (decomp.). The p-compd. gives some of the hexanone and hexene, and a little p-methylcyclohexylamine, b. 148-50°, and dip-methylcyclohexylamine, b. about 265° (decomp.). Menthone oxime, from 1-menthone, cryst. powder, m. 59°, decomp. about 240°,  $[\alpha]_D$  -42.51°, gives menthylamine, menthone and dimenthylamine, b. 305°; hydrochloride, m. about 207° (decomp.). Therefore, besides the normal reaction leading to the formation of primary and secondary amines, the H<sub>2</sub>O formed decomposes part of the oxime into the ketone, and the primary amine formed is also further decomp. into NH<sub>2</sub> and an unsatd. hydrocarbon, the latter being partially reduced to the satd. compd.

C. A. ROUILLER.

Reduction by means of Precipitated Palladium and Sodium Hypophosphite. PIERRE BRETEAU. Bull. soc. chim., 9, 515-7.—The Pd pptd. from a neutral, acid or alk. soln. of its chloride by means of NaH<sub>2</sub>PO<sub>2</sub> can oxidize unlimited amts. of NaH<sub>2</sub>PO<sub>3</sub> in the cold and the H set free can be used for various reduction purposes, even in alc. soln.; an HCl soln. of PdCl<sub>2</sub> is neutralized with Na<sub>2</sub>CO<sub>3</sub> and a satd. soln. of NaH<sub>2</sub>PO<sub>3</sub> is added drop by brop, when a black spongy ppt. is formed with tumultuous evolution of H. If the reduction is to be carried out in acid soln. it is sufficient to keep on adding NaH<sub>3</sub>-PO<sub>2</sub>; if in neutral soln., o.5 mol. Na<sub>2</sub>CO<sub>3</sub> per mol. NaH<sub>2</sub>PO<sub>3</sub> is used; while for alk. reductions an excess of Na<sub>2</sub>CO<sub>3</sub> is added. By this means phenanthrene gives 25-40% tetrahydrophenanthrene without a trace of the dihydro compd., while PhNO<sub>3</sub>, nitronaphthalene and dinitrobenzene and -toluene are reduced to the amines. C. A. R.

Reduction by means of Nickel and Sodium Hypophosphite. PIERRE BRETEAU.

Bull. soc. chim., 9, 518-9.—Amorphous Ni, prepared by adding, in 1 portion, 70 g.

NaH<sub>2</sub>PO<sub>2</sub> to 20 g. NiSO<sub>4</sub> in 100 g. H<sub>2</sub>O on the H<sub>2</sub>O bath, reduces nitro compds. to the amines, but has no action on phenanthrene in neutral soln. (cf. preceding abstr.).

Chas. A. ROUILLER.

Reduction by Calcium and Absolute Alcohol. PIERRE BRETEAU. Bull. soc. chim., 9, 585-6.—Phenanthrene is reduced exclusively to the tetrahydro compd. by pouring 5 g. in 200 cc. abs. alc. upon 15 g. Ca filings, bringing to the b. p., passing in dry NH<sub>2</sub>, removing the flame as soon as the reaction starts, and when the Ca is completely dissolved b. for 1 hr. (cf. Doby, Z. anorg. Chem., 35, 93). C. A. ROUILLER.

Basic Properties of the Oxygen of Ethers. D. E. TSAKALOTOS. Athens, Chem. Lab. Univ. Bull. soc. chim., 9, 519-23.—The viscosities and d. of the systems AcOH-Et<sub>2</sub>O and CCl<sub>2</sub>CO<sub>2</sub>H-Et<sub>2</sub>O at 18° were detd. While the curves representing the values for the first system are normal, i. e., similar to those for systems whose components do not combine, the viscosity curve of the second resembles, in part, that for the system nicotine-H<sub>2</sub>O (C. A., 3, 1954) and the d. curve is like that of the system CHCl<sub>2</sub>-Et<sub>2</sub>O (C. A., 5, 421). The difference in behavior towards Et<sub>2</sub>O of acids having analogous constitutions is explained as being due to the fact that the former reacts like a weak base, forming mol. compds. with strong acids but not with weak ones.

CHAS. A. ROUILLER.

Sparteine. XX. Preparation of Isosparteine. Charles Moureu and Amand Valeur. I'ull. soc. chim., 9, 468-70. XXI. Case of Nitrogen Stereoisomerism. Ibid., 470-2. XXII. Action of Methyl Iodide on Isosparteine. Ibid., 472-6. XXIII. Decomposition of α'-Methylisosparteinium Hydroxide. Ibid., 476-8. XXIV. Methylisosparteine. Ibid., 478-9; cf. C. A., 5, 1589, 2093. Chas. A. Rouiller.

Pyrogenic Decomposition of Metallic Zanthates. ALEXANDRE HÉBERT. Bull. soc. chim., 9, 523-32; cf. C. A., 5, 2094.—The amts. of gas liberated per 100 g. salt should be 4-10 l., and not cc. as stated in the abstr. referred to.

CHAS. A. ROUILLER.

Salts and Esters of the Alkylaminodithiocarbonic Acids. M. E. FOURNEAU. Bull. soc. chim., 9, 532-6.—Ethylmethylaminoacetate ethylmethylaminoacetatedithiocarbonate, EtO<sub>2</sub>CCH<sub>2</sub>NMeC(: S)SNH<sub>2</sub>MeCH<sub>2</sub>CO<sub>2</sub>Et, obtained by slowly adding 25 g. CS<sub>2</sub> in 50 g. Et<sub>2</sub>O to 60 g. MeNHCH<sub>2</sub>CO<sub>2</sub>Et in 20 g. Et<sub>2</sub>O, hexagonal tables, m. 77°; with HgCl<sub>2</sub> it forms mercury ethylmethylaminoacetatedithiocarbonate, (EtO<sub>2</sub>CCH<sub>2</sub>NMeCS<sub>2</sub>)<sub>2</sub>Hg, yellow needles, m. 148°. Mercury propylethylaminoacetatedithiocarbonate, yellow

needles, m. 86°. From a 30% NaOH soln. of these Hg salts, 10 vols. alc. ppt. mercury sodiummethylaminoacetatedithiocarbonate, (NaO<sub>2</sub>CCH<sub>2</sub>NMeCS<sub>2</sub>)<sub>2</sub>Hg, finely divided yellow powder, which is very unstable.

Chas. A. Rouiller.

Phenoxthene. E. FERRARIO. Lab. Org. Inorg. Chem., Geneva. Bull. soc. chim., 9, 536-7.—Ph<sub>2</sub>O (16 g.), S (7 g.) and AlCl<sub>2</sub> (14 g.), heated 14 hrs. on the H<sub>2</sub>O bath, give a good yield of phenoxthene. Metallic Cu, at 250°, converts it into diphenylene oxide.

Chas. A. Rouiller.

Constitution of Indirubin. A. Wahl, and P. Bagard. Bull. soc. chim., 9, 546-8.

—Answer to Maillard (C. A., 5, 2095).

C. A. R.

Negative Case of Indigoid Condensation. ELIE E. PISOVSCHI. Univ. Jassy. Bull. soc. chim., 9, 548-9.—While 2-nitroveratric aldehyde easily undergoes the Baeyer and Drewsen reaction with MeAc in the presence of an alk. (Ber., 15, 2886), giving the corresponding indigo, the 6-nitro compd. is unaffected. Chas. A. ROUILLER.

Bromination of Cyclohexane. F. Bodroux and F. Taboury. Bull. soc. chim., 9, 592-4.—At its b. p. (81°), C<sub>6</sub>H<sub>12</sub> reacts extremely slowly with Br in diffused light, but in direct sunlight 84 g. treated with 160 g. Br, in small portions, each portion being added after complete decolorization to the liquid, gave 90 g. of C<sub>6</sub>H<sub>11</sub>Br. In ultraviolet light there was no reaction. C<sub>6</sub>H<sub>11</sub>Br (75 g.), treated with 75 g. Br at 162°, gave 1,2(?)-dibromocyclohexane, b<sub>100</sub> 144-5°. Chas. A. Rouiller.

Bromination of some Hydroaromatic Compounds in the Presence of Aluminium Bromide. F. Bodroux and F. Taboury. Bull. soc. chim., 9, 595-601.—The compd. to be brominated is dropped in small portions into 100 g. of Br containing 1 g. of Al and allowed to stand several hours at room temp.  $C_0H_{12}$  and  $C_0H_{12}$  Br gave viscous products from which no definit compound could be isolated.  $C_0H_{12}$ Br and cyclohexene both yielded  $C_0Br_0$  quantitatively;  $MeC_0H_{12}$  and the methylcyclohexenes formed  $MeC_0Br_0$ . 1,3- $Me_2C_0H_{10}$  formed  $Me_2C_0Br_0$  (apparently a mixt. of the o-, m- and p-isomers). Menthene and thymomenthene gave only gummy products. C. A. R.

Hydroxyindazoles. P. FREUNDLER. Lab. 2<sup>e</sup> année, inst. chim. appl. fac. sci., Paris. Bull. soc. chim., 9, 601-5; cf. C. A., 4, 1038, 2098. Chas. A. ROUILLER.

Chloro- and Bromoanthranilic Esters. P. FREUNDLER. Bull. soc. chim., 9, 605-8; cf. C. A., 4, 2098.—The following is not reported in the earlier paper: 30 g. o-MeO<sub>2</sub>CC<sub>4</sub>H<sub>4</sub>NH<sub>2</sub> and 35 g. anhydrous CCl<sub>2</sub>CHO, treated with 31 g. Br and allowed to stand 12 hrs., give, 30 g. of a mixt. of methyl 5-bromoanthranilate, yellowish white prisms, m. 74°, slightly sol. in cold MeOH, and methyl-3,5(?)-dibromoanthranilate, needles, sm. 84°, sol. in alc.

CHAS. A. ROUILLER.

Hydrazones of Phenacylamines. Max Busch and Georg Hefele. J. prakt. Chem., 83, 524-53.—With the hope of obtaining a direct proof of the stereoisomerism of hydrazones, the evidence for which at present is based chiefly on analogy with the stereoisomerism of the oximes, the authors have prepared hydrazones of aminoketones of the type ArCOCH<sub>2</sub>NHR, the syn forms of which, by intramol reaction of the hydrazino- and the amino- (of substituted amino-) groups, might be expected to undergo ring closure. Many aminoketones of the preceding type have been prepared, but only in the case of the  $\psi$ -cumidine deriv., BzCH<sub>2</sub>NHC<sub>2</sub>H<sub>11</sub>, have 2 forms of the phenylhydrazone been isolated; unfortunately, 1 form has been obtained in amt. so small that the special purpose of the research has not been fulfilled. The phenylhydrazones condense with aldehydes to form tetrahydrotriazines, and are readily oxidized by an excess of the hydrazine or by CrO<sub>2</sub> to dihydro-1,2,3-triazoles, which are reduced by Na and alc. to tetrahydrotriazoles. The primary purpose of the research has not been accomplished by an exam. of the semicarbazones and thiosemicarbazones of the preceding aminoketones; the semicarbazones show a tendency to yield

ketotetrahydrotriazines. Only with aminoketones of the type COPhCH<sub>2</sub>NR<sub>2</sub> have 2 forms of the phenylhydrazone been obtained; in these cases, unfortunately, the configurations cannot be detd. by the authors' methods, because ring closure cannot occur. Phenylacylamines, PhOCCH<sub>2</sub>NHR, are readily obtained by the interaction of w-bromoacetophenone and an amine (2 mols.) in alc. The hydrazones are obtained in about 80% yield by adding a small excess of the hydrazine to a suspension of the aminoketone in cold alc. containing a little AcOH and also H<sub>2</sub>S to prevent the oxidation of the hydrazone to the dihydrotriazole. The following new compds. have been prepared by the preceding methods. w-Benzylaminoacetophenone forms a phenylhydrazone, PhHNN: CPhCH<sub>2</sub>NHCH<sub>2</sub>Ph, m. 76°, an alc. soln. of which is converted by b. glac. AcOH into a substance, m. 176°; it is probably the dihydrotriazole. With PhN<sub>2</sub>H<sub>2</sub>, w-anilinoacetophenone yields 1,2,4-triphenyl-2,5-dihydro-1,2,3-triazole, (I), m. 136.5°, while with semicarbazine and thiosemicarbazine it yields the semi-

carbazone, m. 171° (decomp.), and the thiosemicarbazone, m. 167° (decomp.); the former forms a hydrochloride, m. 230° (decomp.), and at 200° loses NH<sub>8</sub>, yielding 2-keto-1,5-diphenyl-1,2,3,6-tetrahydro-1,3,4-triazine (II), m. 181° (decomp.). ω-p-Toluidinoacetophenone forms a phenylhydrazone, C<sub>m</sub>N<sub>m</sub>N<sub>8</sub>, m. 147°, which reacts with phenylcarbamide on the H<sub>2</sub>O bath to form a carbamide, PhNHCONPhN: CPhCH<sub>2</sub>-NHC<sub>8</sub>H<sub>8</sub>Me, m. 184°, and with BzCl in C<sub>8</sub>H<sub>8</sub>N, yielding a benzoyl derivative, PhNBzN: CPhCH<sub>2</sub>NHC<sub>8</sub>H<sub>8</sub>Me, m. 110°. The preceding phenylhydrazone reacts with BzH at 120° and with salicylaldehyde at 150° to form substances which are probably tetrahydrotriazines; they have various m. ps. (decomp.) on account of their property of retaining variable quantities of different solvents. The phenylhydrazone is also readily converted into 2,4-diphenyl-1-p-tolyl-2,5-dihydro-1,2,3-triazole (III), m. 152° which does not react with phenylcarbamide, aldehydes, HNO<sub>2</sub>, or BzCl, and is reduced by Na and alc. to the tetrahydrotriazole, C<sub>m</sub>H<sub>m</sub>N<sub>8</sub>, m. 122°. ω-p-Toluidinoace ophenone forms a semicarbazone, C<sub>16</sub>H<sub>10</sub>ON<sub>4</sub>, m. 182° (decomp.), which evolves NH<sub>2</sub> at 205° and yields the 2-keto-5-phenyl-1-p-tolyldihydro-1,3,4-triazine (IV), m.

208°. ω-o-Anisidinoacetophenone, MeOC<sub>2</sub>H<sub>4</sub>NHCH<sub>2</sub>Bz, yellow needles, m. 89°, forms a semicarbazone, C<sub>10</sub>H<sub>11</sub>O<sub>2</sub>N<sub>4</sub>, m. 176° (decomp.), and a phenylhydrazone, C<sub>20</sub>H<sub>11</sub>ON<sub>2</sub>, m. 105°; from the latter is obtained the 2,4-diphenyl-1-o-anisyldihydro-1,2,3-triazole (V), m. 151.5°. The reaction between ω-bromoacetophenone and p-anisidine yields 3 products: ω-p-anisidinoacetophenone, m. 93°, yellow leaflets (which differ from all the other phenacylamines by giving a violet coloration with alc. FeCl<sub>2</sub>), a substance m. 181°, orange-yellow plates, and diphenacyl-p-anis dine, MeOC<sub>2</sub>H<sub>4</sub>N(CH<sub>2</sub>COPh)<sub>2</sub>, m. 180°, yellow needles. ω-p-Anisidinoacetophenone forms a semicarbazone, m. 145° (decomp.), and an oily phenylhydrazone from which the corresponding dihydrotriazole, C<sub>10</sub>H<sub>12</sub>ON<sub>2</sub>, m. 156°, is obtained; ω-p-chloroanilinoacetophenone forms a semicarbazone, C<sub>10</sub>H<sub>12</sub>ON<sub>4</sub>Cl, m. 167° (decomp.), and a phenylhydrazone, m. 147°, from which 2,4-diphenyl-1-p-chlorophenyldihydro-1,2,3-triazole, C<sub>20</sub>H<sub>16</sub>N<sub>3</sub>Cl, m. 153°, is obtained. ω-φ-Cumidinoacetophenone, Me<sub>2</sub>C<sub>2</sub>H<sub>2</sub>NHCH<sub>2</sub>COPh, yellow needles, m. 122°, forms a semicarbazone, m. 179° (decomp.), and a phenylhydrazone, m. 155°; from the mother liquor in the latter preparation a very small amt. of a yellow substance is some-

times obtained, which crystallizes in rhombohedra, and shows the reactions of a hydrazone. From the phenylhydrazone, m. 155°, is obtained 2,4-diphenyl·1-\$\psi\$-cumyl-dihydro-1,2,3-triazole, C22H2N1, m. 139°. \$\omega\$-Dibenzylaminoacetophenone yields 2 isomeric \$phenylhydrazones\$, which are separated by alc.; the less stable form crystallizes in colorless leaflets, m. 107°, the more stable in needles, m. 75°. The latter change to the former at 105°, or by treating an alc. soln. of the needles with HCl and decomp. the resulting \$hydrochloride\$, m. 255° (decomp.), with NH2. \$\omega\$-Phenylmethylaminoacetophenone forms a \$phenylhydrazone\$, C2H2N2, m. 98°, and a semicarbazone\$, C16H18ON4, m. 202° (decomp.). \$\omega\$-Phenylethylaminoacetophenone forms, in the absence of air, 2 isomeric semicarbazones\$, C17H20ON4, 1, m. 153°, crystallizing in leaflets, the other m. 145°, in needles.

Aliphatic Nitro Compounds. X. Hydroxamyl Chlorides. WILHELM STEINKOPF AND BORIS JURGENS. Chem. Inst. Techn. Hochsch., Karlsruhe. J. prakt. Chem., 83, 453-70.—A continuation of C. A., 5, 1766. The substance previously recorded as  $\beta$ -oximino-oxaliminochloride (see C. A., 4, 2278) from its reactions and methods of formation, may be chloro-oximinoacetamide, produced ex. gr., from nitroacetonitrile

and HCl in dry Et,O, by the following series of reactions: O,NCH,CN-→HON :  $C(OH)CN \longrightarrow HON : CCI = CN + H_2O \longrightarrow HON : CCICONH_2$ . At first sight the explanation appears improbable, because it is unlikely that the H<sub>2</sub>O formed in the second reaction can convert the nitrile into the amide in the presence of a large excess of Et.O. However, the discovery that HCl converts Et nitroacetate in dry Et<sub>2</sub>O into Et chloro-oximinoacetate shows that the first two phases of the series above can occur. The fact that the third phase does occur, and that the so-called  $\beta$ -oximinooxaliminochloride is really chloro-oximinoacetamide, is proven by the action of aqueous Na<sub>2</sub>CO<sub>3</sub> on the substance in the cold, whereby an odor of a nitrile oxide is observed, and furoxandicarboxylamide (succinamidedinitrosoperoxide) is formed. analogy to the preceding, the " $\alpha$ -oximino-oxaliminochloride," m. 173-4° (decomp.), produced from methazonic acid and HCl in Et.O (loc. cit.), must be chloro-oximinoacetaldoxime (chloroglyoxime), HON: CCICH: NOH. This is so, because not only is the substance converted by SOCl, in dry Et,O into chloro-oximinoacetonitrile, CNCCl: NOH (which is too unstable to be analyzed, but yields with PhNH, anilino-oximinoacetonitrile, PhNHC(CN): NOH, m. 138-9°, but also the identity of the substance with Hantzsch's chloroantiglyoxime, m. 161° (the m. p. can be raised to 168° by repeated crystallization), is proved by the fact that the 2 substances form the same acetyl deriv., m. 163-4°, and diacetyl deriv., m. 90.5°. Moreover, a moist Et<sub>2</sub>O soln. of the chloro-oximinoacetonitrile is hydrolyzed by HCl, yielding chloro-oximinoacetanilide Chloro-oximinoacetic acid, HON: CClCO, H, decomp. 129°, is obtained by keeping for three weeks a suspension of K nitroacetate in dry Et,O satd. with HCl in the cold. In Nageli's chloroamphiglyoxime the Cl at. and I of the OH groups have the anti-configuration, because the substance is insensitive to alkalis and does not yield a nitrile oxide with Na<sub>2</sub>CO<sub>2</sub>. An aqueous soln. of this antichloroamphiglyoxime reacts with PhNH<sub>2</sub> to form a salt, HON: CCICH: NOH, PhNH<sub>2</sub>, m. 114° (decomp.). This behavior not only illustrates the stability of the halogen at. in antihydroxamyl chlorides, but may also serve for the characterization of such substances, because other hydroxamyl chlorides, in which the Cl at. and the OH group have the syn-configuration react with PhNH, to form anilino derivs. by substitution of the halogen at. The authors deduce from the preceding that the  $\alpha$ - and the  $\beta$ -oximinohydroxamic acids previously described (loc. cit.) have the anti- and the syn-configurations, resp. By chlorinating a cold soln. of chloroantiglyoxime in dil. HCl, antidichloroglyoxime  $(\alpha, \beta-dichloro-\alpha, \beta-dioximinoethane)$ , HON: CCl = CCl: NOH, m. 201°, is obtained,

which yields the odor of a nitrile oxide by heating, or, by treatment with Na<sub>2</sub>CO<sub>3</sub>, forms a dibenzoyl derivative, m. 217°, and reacts with PhNH<sub>2</sub> to form α,β-dianilino-α,β-dioximinoethane. Ethyl iodo-oximinoacetate, EtOCCI: NOH, m. 95-6°; iodo-oximinoacetamide, HON: CICONH<sub>2</sub>, yellow crystals, decomp. 154-5°; iodoantigly-oxime, HON: CICH: NOH, m. 136°; and antidiiodoglyoxime, HON: CICI: NOH, decomp. 172°, are obtained by heating the corresponding Cl compds. with a soln. of NaI in Me<sub>2</sub>CO. Antichloroamphiglyoxime does not react with NaI, in Me<sub>2</sub>CO; this is another instance of the stability of the Cl at. in antichloro-oximino compds.

B. F. PARLETT BRENTON.

Constitution of Thiophenoquinones and Mechanism of Quinone Reactions. Theo-DOR POSNER. Chem. Inst., Univ. Greifswald. J. prakt. Chem., 83, 471-83.—Definitive reply to Michael (C. A., 4, 898) and to Michael and Cobb (C. A., 5, 474). B. F. PARLETT BRENTON.

Humulene from Hop Blossom Oil. E. Beckmann and Ernst Deussen. J. prakt. Chem., 83, 483-9.—Humulene,  $b_{10}$  118-9°, isolated from oil hop flowers by fractional distillation, has been identified with iso- $\alpha$ -caryophyllene by a comparison of the nitrosates and nitrosochlorides.

B. F. Parlett Brenton.

Templin Oil. ERICH BOCKER AND ALFRED HAHN. J. prakt. Chem., 83, 489-98. —The oil of the dwarf pine (Pinus pumilo), freed from terpenes and sesquiterpenes, d18 0.8707, [a] -9° 45', esterification no. 13.4, has been examined with respect to its oxygenated constituents. In addition to l-bornyl acetate, it contains aldehydic and ketonic substances, and at least 30% of alcs. and esters of the terpene and sesquiterpene series, as yet unexamined. The oil has been sepd. into 12 fractions bis between 85° and 178°. The fractions are dissolved separately in 96% alc., and shaken for 2-3 hrs. with 30% aq. NaHSO<sub>2</sub>. The fraction  $b_{11}$  148-60°,  $[\alpha]_D$  -14° 15', esterification no. 53, gives by this treatment a small yield of an H sulfite compd., which after hydrolysis yields an oil, C1. HanO. This is unsatd. and restores the color of Schiff's reagent. The fractions b<sub>12</sub> 138-48°, and 127-38° are united and distilled; the portion b<sub>18</sub> 128-35° yields an H sulfite compd., from which, by hydrolysis, is obtained a substance, C10H20; which is l-rotatory, unsatd., and probably of ketonic nature. The fractions b<sub>12</sub> 105-9°, 109-113°, 113-8°, and 118-27° do not yield H sulfite compds., but contain *l*-bornyl acetate. The fractions  $b_1$ ,  $85-100^{\circ}$  and  $100-5^{\circ}$  differ from all the others by having an intense peculiar odor. By combining them and distilling, a portion b<sub>14</sub> 87-95° is obtained, which yields a white, cryst. H sulfite compd. The substance C<sub>8</sub>H<sub>14</sub>O, obtained by the hydrolysis of this compd. by 20% Na<sub>2</sub>CO<sub>3</sub>, has ketonic properties, and is called pumilone, b<sub>784</sub> 216-7°, d<sup>18</sup> 0.9314, d<sup>20</sup> 0.9288, n<sub>D</sub> 1.46459, and  $[a]_{0}$  —15°. It has a very intense, not unpleasant odor. The odor of the natural dwarf pine oil is mainly due to pumilone, altho it is present only to the extent of 1-2%. Pumilone, which is unsatd. and contains I double linking, does not yield characteristic derivs., except the H sulfite compd. and the semicarbazone, m. 116-7°.

B. F. PARLETT BRENTON.

o-Hydroxyquinoline. Georg Cohn. J. prakt. Chem., 83, 498-506.—8-Hydroxyquinoline and HCHO, which yield hydroxymethyl-8-hydroxyquinoline under Manasse's conditions, produce another substance called "new hydroxyquinolinecarbinol," when a mixt. of 5 g. of 8-hydroxyquinoline, 15 cc. of formalin, and 10 cc. of 20% NaOH are heated on the H<sub>2</sub>O bath; by diluting with H<sub>2</sub>O and neutralizing with HCl or AcOH, the new compd., C<sub>12</sub>H<sub>2</sub>O<sub>2</sub>N, is obtained as a yellow, amorphous powder. It does not m. at 250°, evolves formaldehyde at higher temps., forms a soln. in dil. HCl which is colored dark green by FeCl<sub>3</sub>, yields a yellow Na salt, couples with diazobenzenesulfonic acid, and is oxidized by alkaline K<sub>2</sub>Fe(CN)<sub>6</sub> to a dark green substance. 8-Hydroxyquinoline-5-sulfonic acid is formed when loretin (7-iodo-8-hydroxyquinoline-5-sul-

fonic acid) is boiled with H<sub>2</sub>O and PhNH<sub>2</sub> or phenetidine, with piperidine, or with guaiacol and NaOH. 5-Nitroso-8-hydroxyquinoline is reduced by PhN<sub>2</sub>H<sub>2</sub> on the H<sub>2</sub>O bath, yielding 5-azo-8-hydroxyquinoline, HOC<sub>2</sub>NH<sub>2</sub>N: NC<sub>2</sub>NH<sub>2</sub>OH, m. 220° (decomp.), brownish red needles with a blue reflex. By reduction with K<sub>2</sub>SO<sub>3</sub> the nitroso compd. yields a substance, not yet fully examined, which is probably an aminohydroxyquinolinesulfonic acid.

B. F. Parlett Brenton.

Mechanism of the Elimination of Halogens by Aromatic Amines. I. OSTROM-ISSLENSKII AND P. ALABYEV. J. prakt. Chem., 83, 506-12.—Hitherto an explanation has not been attempted of the fact that certain aromatic amines (PhNH., PhN.H., C<sub>n</sub>H<sub>a</sub>N, C<sub>o</sub>H<sub>3</sub>N) eliminate the halogen from various olefin dihalogenides with the regeneration of the original olefin. Thus the authors show that when stilbene dibromide is heated with dry C, H, N for 12 hrs. on the H,O bath, about 87% of the halogen is eliminated as HBr, stilbene itself being formed. Consequently, in such reactions, the H of the halogen acid must have been withdrawn from the aromatic amine and, furthermore, must have been withdrawn from the aromatic nucleus, since the elimination in question can be effected by tertiary amines. It follows, therefore, that unstable groups, such as C.H.NH, must be formed. From these groups by intramol. change, polymerization, etc., probably are derived the resinous substances which are almost invariably produced in the reactions in question. The formation of benzidine itself has never been observed in such reactions. The author's explanation accounts for the formation of tetramethyldiaminodiphenylethane observed by Schoop when dimethylaniline and ethylene dibromide are heated for 8 days on the H<sub>2</sub>O bath.

B. F. PARLETT BRENTON.

Action of Light on Lactates and Lactic Acid. D. Ganassini. Ist. fisiol. Pavia. Seduta soc. med. chir., Pavia, 1909; thru Zentr. Biochem. Biophys., 11, 378.—The earlier work showing that lactic acid in light and atm. O forms CO<sub>2</sub>, pyruvic acid and AcH is extended to show that the salts act similarly, some (Fe, Cu, and Hg), even in absence of light. Finally, it was found that d-lactic acid produces AcH and l-acid produces only pyruvic acid. Hence the earlier result mentioned above is explained as due to the fact that the dl-acid was used.

I. K. Phelps.

Ammonium Cyanate and Urea. R. ESCALES. Chem. Ztg., 35, 595.—It was found that urea, heated in vacuo at 160-80°, is converted into NH<sub>2</sub>CON, which sublimes. If Wöhler had purified his synthetic urea by vacuum distillation, his epochmaking discovery would never have been made.

M. Heidelberger.

Action of Ultraviolet Rays on Saccharose. H. BIERRY, V. HENRI AND A. RANC. Compt. rend., 152, 1629-32.—An inversion of cane sugar soln. (5%) is observed after 20 hrs. exposure to ultraviolet rays at 40°. After 48 hrs. HCOH can be detected and later an evolution of gas, containing CO. The gas formation does not occur in the presence of CaCO<sub>2</sub>.

ISRAEL S. KLEINER.

Action of Hypoiodous Acid on Unsaturated Acids. α-Cyclogeranic Acid. J. BOUGAULT. Ann. chim. phys., 22, 125-36; Chem. Zentr., 1911, I, 1056; cf. C. A., 4, 1480.—Cyclogeraniolene nitrosate does not m. 102-4°. Its m. p. depends much on the quickness of heating and can reach 115°. The author obtained another nitrosate m. 117° which yields a tri-Me-cyclohexenone oxime m. 59°. E. J. CRANE.

Quinine and Euquinine. A. ASTRUC AND L. COURTIN. J. pharm. chim., 3, 292-4.
—Quinine being dibasic and euquinine monobasic, the authors suggest that the CO<sub>2</sub>Et may be linked to the piperazine N making it pentavalent instead of being linked, as heretofore supposed, to the C at. connecting the 2 rings.

V. K. CHESNUT.

Preparation of Mentholglucuronic Acid. I. BANG. Univ. Lund. Biochem. Z., 32, 445.—The method of Neuberg and Lachman (C. A., 4, 1765) can be simplified. Urin

is 0.5 satd. with (NH<sub>4</sub>)<sub>x</sub>SO<sub>4</sub>, brought to the b. p. and filtered hot. On cooling the NH<sub>4</sub> mentholglucuronate separates.

G. M. MEYER.

Synthesis of Polypeptides. Derivatives of  $\alpha$ -Aminobutyric Acid and their Behavior towards Peptolytic Ferments. Emil Abderhalden, Hsing Lang Chang and Emil WURM. Tierarztliche Hochschule, Berlin. Z. physiol. Chem., 72, 24-37.—dl-α-Aminobutyric acid was prep. according to Fischer and Mouneyrat's method. Brucine formyldl-aminobutyrate was used for the separation of the optical isomers. d-Aminobutyric acid  $[\alpha]_{\rm p}^{20^{\circ}}$  8.12°. l-Aminobutyric acid  $[\alpha]_{\rm p}^{20^{\circ}}$  -7.86°. Chloracetyl-dl-aminobutyric acid, prep. by the action of chloracetyl chloride on dl-aminobutyric acid; yield 80%, m. 130° (cor.). Pointed platelets from AcOEt + petroleum ether. Glycyl-dl-aminobutyric acid from the chloracetyl deriv. and 25% NH<sub>2</sub>. Yield, 70%. Cryst. from 50% alc., m. 231° (cor.). Chloracetyl-d-aminobutyric acid  $[\alpha]_D^{20}$  -18.14°. Prep. like dl-acid; m. 119°, softens 112°; fine needles from abs. alc. and petroleum ether. Glycyl-d-aminobutyric acid,  $[\alpha]_{p}^{20^{\circ}}$  —12.24°; prep. like dl-comp. Yield, 72%; m. 223° (cor.), becomes yellow 215°. Chloracetyl-l-aminobutyric acid [α]<sub>p</sub><sup>20°</sup> 19.0°, m. 119°; long needles from AcOEt + petroleum ether. Glycyl-l-aminobutyric acid  $[a]_{p}^{20^{\circ}}$ 18.29°, m. 222°, yellow at 215°. G. M. MEYER.

Methylated Polypeptides. EMIL ABDERHALDEN AND KARL KAUTZSCH. Tierārz. Hochschule, Berlin. Z. physiol. Chem., 72, 44-50.—dl-Leucyl-glycine is dissolved in N KOH (1 mol.) and MeOH. MeI is added from time to time in small portions till 3 mols. have been added. After standing the soln. is pptd. with 10% phosphotungstic acid and the ppt. decomp. with Ba(OH)<sub>2</sub>. After freeing the sol. from Ba and conc. in vacuo the trimethylleucylglycine chloroplatinate separates as orange-red prisms or platelets. Chloroaurate, m. 170-2°. Picrate lemon-yellow prisms from alc., m. 225-6°; decomp. 240-50°.

G. M. Meyer.

Derivatives of Amino Acids. IV. Combination with Glycerol. Emil. Abder-Halden and Louis Baumann. Tierärztliche Hochschule, Berlin. Z. physiol. Chem., 72, 50-7.—Glyceromonotyrosine ether gives a red coloration with Millon's reagent even in the cold. This is not due to the presence of tyrosine as a probable impurity, because the substance prepared from the Cu salt behaves similarly. m-Cresolglycerol ester m. 65-70°; long narrow rectangular plates. Gives the cresol reaction with FeCl<sub>2</sub>. Chloro-m-cresolglycerol ester m. 90°; bushy needles from  $C_0H_0$ . Glycerodilyrosine ether was prep. by the action of  $\alpha,\alpha$ -dichlorohydrin on l-tyrosine in alc. EtONa; would not cryst. Red color with Millon's reagent on warming. Obtained amorphous thru its Cu salt, m. 275°. The substance could not be esterified with MeOH. Glycerodi-(glycyl-l-tyrosine) ether, m. 247°, with Millon's a white ppt., on warming the red color appears. Glycerotrityrosine ether, m. about 295°. Ethyl ester hydrochloride, m. 83°. G. M. Meyer.

Synthesis of  $\alpha$ -Amino Ketones by means of Hexamethylenetetraamine. C. Mannich and Priedrich L. Hahn. Pharm. Inst., Univ. Berlin. Ber., 44, 1542-52.—A number of ketones with halogen in the  $\alpha$ -position form additive compds., RCOCH<sub>2</sub>N<sub>4</sub>(CH<sub>2</sub>)<sub>6</sub>X, (X = halogen) with hexamethylenetetraamine. The substances of this class, described below, are all crystallin and have the character of salts, the halogen being ionized in aq. soln. They decomp. slowly at the ordinary temp., more quickly when heated, so that approximately constant m. ps. can be obtained only by raising the temp. rapidly. The additive compds. are hydrolyzed to  $\alpha$ -amino ketones by the action of alc. (8 pt.) and aq. HCl (1 pt.) (38%), at the ordinary temp. The additive compds. were obtained from the following ketones.  $\omega$ -Chloroacetophenone, m. 145°. Yield, 60%.  $\omega$ -Bromoacetophenone, m. 165°. Yield, 80%. p-Methoxy- $\omega$ -chloroacetophenone, m. 170°. Yield, 50%.  $\omega$ -Chloroacetopyrocatechyl diacetate (see below). Yield, 40%.  $\omega$ -Iodoacetopyrocatechyl diacetate (see below).

veratrone (see below), needles. Yield, practically quant. ω-Iodoacetopyrogallyl triacetate (see below), m. about 130°. Chloroacetone, lustrous needles, m. 122°. Yield, poor. Iodoacetone, m. 146°. Yield, quant. No additive compds. could be obtained from ω-bromoacetopyrogallyl trimethyl ether, or from ω-chloroacetopyrogallyl triacetate. The yield of w-aminoacetophenone hydrochloride, from the above compd. of w-chloroacetophenone is about 35% of the additive compd. From the bromo deriv. the yield of amino ketone hydrobromide is about 21% of the additive compd. ω-Carbethoxyaminoacetophenone, BzCH,NHCO,Et, is prepared from a salt of the amino ketone, Et chlorocarbonate and aq. NaHCO3, at the ordinary temp.; slender needles, m. 58°. It is not decomp. by conc. H<sub>2</sub>SO<sub>4</sub>; alc. and Na-Hg (5%) reduce it to ω-carbethoxyaminomethylphenylcarbinol, HOCHPhCH\_NHCO\_Et; white scales from AcOEt + petroleum ether, m. 86°. p-Methoxy-w-aminoacetophenone hydrochloride, MeOCaH4-COCH<sub>2</sub>NH<sub>2</sub>.HCl, from the additive compd. as above; crystallin, m. 197° (decomp.). At 160-70°, ag. HCl (38%) hydrolyzes it to b-hydroxy-w-aminoacetophenone, HOC. H4-COCH, NHa; lustrous, stable plates. Hydrochloride, white plates from alc., m. 242° (decomp.). φ-ω-Dicarbethoxy-ω-aminoacetophenone, EtO,COC,H,COCH,NHCO,Et, from the preceding compd., Et chlorocarbonate and aq. NaOH; white plates, m. 85-6°. w-Chloroacetopyrocatechol, (HO), CaH, COCH, Cl, is prepared by heating on a b. H<sub>2</sub>Obath, until HCl evolution ceases, pyrocatechol, chloroacetic acid and POCl, (equal pts.); crystals from H<sub>2</sub>O, m. 173°. Diacetyl derivative, from Ac<sub>2</sub>O + conc. H<sub>2</sub>SO<sub>4</sub> (2 drops), at the ordinary temp.; Crystals from glacial AcOH m. 110-1°. Yield, almost quant. With 1 N NaI, in acetone, it gives  $\omega$ -iodoacetopyrocatechyl diacetate; crystals from alc. (50%), m. 110-1°. Yield, quant. ω-Aminoacetopyrocatechol, from the additive compd.; decomp. without m. above 200°. Hydrockloride, lustrous plates from H<sub>2</sub>O, darkens 230°, m. 252°. At the ordinary temp., in CHCl2, Br and acetoveratrone give w-bromoacetoveratrone, (MeO), CaH, COCH, Br; crystals from AcOEt, m. 80-1°. Yield, 80%. Its additive compd. forms w-aminoacetoveratrone hydrochloride, (MeO), C, H, COCH, NH, .HCl; white, lustrous needles, m. 221°. Yield, 30% of the additive compd. This hydrochloride is not identical with A. Pictet and A. Gam's "ω-aminoacetoveratrone hydrochloride" (C. A., 3, 2958). Conc. HCl hydrolyzes it to  $\omega$ -aminoacetopyrocatechol hydrochloride. w-Chloroacetopyrogallyl triacetate, (AcO), C, H, COCH, Cl, from w-chloroacetopyrogallol, Ac<sub>2</sub>O and conc. H<sub>2</sub>SO<sub>4</sub>, at the ordinary temp.; crystals from glacial AcOH, m. 100-1°. Yield, almost quant. Its constitution is shown by the fact that, with KMnO4, in acetone, it gives an acetylated acid which is hydrolyzed to 2,3,4-trihydroxybenzoic acid (pyrogallolcarboxylic acid). w-Iodoacetopyrogallyl triacetate, from the Cl deriv. and NaI, in acetone; crystals from AcOEt + ligroin, m. 139-40°. Yield, nearly quant. 2,3,4-Trimethoxyacetophenone, (MeO), C, H, Ac, is prepared from pyrogallyl trimethyl ether, AcCl and AlCla, in CS2, or from acetopyrogallol, Me2SO4 and aq. KOH (50%); crystallin, m. 14-5°; b<sub>13</sub> 165°. With Br and AcONa, in glacial AcOH, it gives w-bromoacetopyrogallyl trimethyl ether; white crystals from Et<sub>2</sub>O, m. 50-1°; b<sub>13</sub> 174-6°. Only traces of KBr are formed when it is b. during 2 hrs. with alc. AcOK, but Br is readily eliminated by b., alc. KOH. The compd. reacts instantly with NaI. It is just possible that the Br is present in the nucleus. Aminoacetone is formed, in small yield, by the hydrolysis of either the chloro- or iodoacetone additive compds. described above. J. BISHOP TINGLE.

Chemical Action of Light. XIX. GIACOMO CIAMICIAN AND P. SILBER. Bologna. Ber., 44, 1280-9; see C. A., 4, 1868.—In addition to isobutyleneglycol, acetone and MeOH, when mixed (1:2) and exposed to light during 1 yr., give  $Pr^{\beta}OH$  and ethyleneglycol, which were separated by distillation. The ethyleneglycol appears to be formed from MeOH and HCHO, which latter is produced as the result of the reduction of the

acetone. Acetone and alc., under similar conditions, give diacetyl (from the AcH),  $Pr^{\beta}OH$ , trimethylethyleneglycol, sym.-dimethylethyleneglycol (isolated after oxidation, as diacetyldioxime), methyl isopropyl ketone and a compound,  $C_0H_{12}O_2$ ; oil, b. 149°. It is stable towards KMnO<sub>4</sub> and does not contain EtO. The portion of reaction product which contains the sym.-dimethylethyleneglycol gives with Ph isocyanate, 2 phenylurethans, which are separated by means of  $C_0H_0$ . The less sol. m.  $201-2^{\circ}$ , the other m. 175°. They are probably stereoisomers. The glycol also forms 2 benzoyl derivatives, 1 is a liquid,  $b_{16}$  217-8°. The second is deposited from petroleum ether in monoclinic crystals, m. 77°; a:b:c 0.4170: 1:0.3337;  $\beta$  69° 6′. A mixture of equal pt. of acetone and  $Pr^{\beta}OH$ , after exposure to light during about 9 mos., gave only the additive product, pinacone,  $C_0H_{14}O_2$ . Methyl ethyl ketone behaves quite differently from acetone. Earlier work with alc. and benzophenone and acetophenone, resp., has been repeated. From alc. and benzophenone the only product was benzopinacone. See following abstrs.

Chemical Action of Light. XX. GIACOMO CIAMICIAN AND P. SILBER. Bologna. Ber., 44, 1554-8; see preceding and following abstrs. and Ber., 34, 1541 (1901).—A mixture of acetone (1 vol.) and Et<sub>2</sub>O (2 vols.), which had been exposed to light during the summer and autumn, was found to contain Pr<sup>6</sup>OH, and 2 compounds, possibly C<sub>1</sub>H<sub>16</sub>O<sub>2</sub>, and C<sub>11</sub>H<sub>26</sub>O<sub>3</sub>, or C<sub>11</sub>H<sub>22</sub>O<sub>3</sub>. The former is probably trimethylethoxyethyl alcohol, HOCMe, CHMeOEt; b. 138-41°. At 130°, dil. H, SO, (1:10) converts it into methyl isopropyl ketone. The second compd. is found in the residue, after steam distilling the original mixture; oil, b<sub>20</sub> 109-12°; mol. wt., in glacial AcOH, 196.6-201.2. Acetophenone (50 g.) and Et<sub>2</sub>O (200 cc.), after exposure to light from May to Feb., give chiefly an additive compound, possibly HOCPhMeCHMeOEt; liquid, b. 247°. The remaining material consists of high b. substances, together with acetophenonepinacone. A mixture of benzophenone (50 g.) and Et<sub>2</sub>O (200 cc.) was exposed to light from June to December. The products consisted of benzopinacone (23 g.), AcH, and a compound, possibly, HOCPh-CHMeOEt; it distils slowly with steam and is sol. in petroleum ether, which properties enable it to be separated from the original mixture; colorless, pearly lustrous prisms from dil. MeOH, m. 51°. The resinous, non-volatil material, which is also produced, contains 14.8% of EtO; mol. wt., in glacial AcOH, 363.7-72.6. With Et<sub>2</sub>O, therefore all 3 ketones form additive compds. of the glycol type, whereas, with alc. in place of Et<sub>2</sub>O, benzophenone is converted completely into benzopinacone. J. BISHOP TINGLE.

Chemical Action of Light. XXI. GIACOMO CIAMICIAN AND P. SILBER. Bologna. Ber., 44, 1558-64; see preceding abstrs. and C. A., 3, 2294.—The resin, obtained by the insolation of BzH, contains a tetrameric compd., Mascarelli's trimeric deriv. and an isomeric trimeric compound, (C<sub>7</sub>H<sub>a</sub>O)<sub>2</sub>, which is separated by its sparing sol. in Et<sub>2</sub>O + petroleum ether and in cold MeOH. Voluminous, white needles from alc. + H<sub>2</sub>O, m. 144-5°; mol. wt., in glacial AcOH, 310.9-12.5. It does not combine with semicarbazine. After exposure to light during more than a year, cinnamic aldehyde gives a compound, CoH1O; amorphous, white powder from Et2O + petroleum ether, softens 103°, m. 115°; mol. wt., in C<sub>6</sub>H<sub>6</sub>, 523-606. In alc. it is immediately oxidized by "permanganate." From a mixture of benzophenone (25 g.) and BzH (58 g.), insolated from June to January, a resin and a compound were obtained, which were separated by means of cold, glacial AcOH, in which the latter is insol.; white, voluminous needles from AcOEt, m. 245°. Its formula is C<sub>21</sub>H<sub>22</sub>O<sub>2</sub>, not C<sub>41</sub>H<sub>34</sub>O<sub>6</sub>, as stated previously. In CaHa, during a year's exposure, benzophenone and di-Et dihydrocoll dinedicarboxylate give benzopinacone and di-Et collid.nedicarboxylate. A soln. of equal pts. of quinaldine and acetone, insolated during a year, give a compound, C22H22N2. It was separated by distilling off the acetone and steam distilling the residue. The nonvolatil portion deposits warty aggregates of slender, yellow needles from C<sub>8</sub>H<sub>4</sub>, m. 212°. In alc. it is oxidized by "permanganate." Dihydrockloride, C<sub>28</sub>H<sub>22</sub>N<sub>2</sub>.2HCl, from dry HCl, in Et<sub>2</sub>O; white needles. Alc. converts it into the monohydrockloride, C<sub>28</sub>H<sub>22</sub>N<sub>2</sub>.HCl; warty aggregates of orange-red crystals, sublimes without m. 270°. Chloroplatinate, C<sub>28</sub>H<sub>22</sub>N<sub>2</sub>.H<sub>2</sub>PtCl<sub>6</sub>, orange-red crystals. Chloroaurate, C<sub>28</sub>H<sub>22</sub>N<sub>2</sub>.HAuCl<sub>2</sub>, brown powder. Its formation is preceded by that of small, golden yellow crystals which become brown in contact with H<sub>2</sub>O.

J. BISHOP TINGLE.

Oxalosuccinic Ester. WILHELM WISLICENUS AND MARTIN WALDMULLER. Chem. Lab., Univ. Tübingen. Ber., 44, 1564-73.—Tri-Et oxalosuccinate, EtO<sub>2</sub>CCOCH-(CO,Et)CH,CO,Et, is best prepared by means of EtOK. Potassium derivative, C1.H., O2K, colorless, slender, interlaced needles from AcOEt. In H.O the soln. is neutral; with FeCl, a brownish red color is produced which disappears almost instantly. A deep red color appears slowly if the K deriv. is treated with acid, extracted immediately with Et.O and quickly mixed with alc. and FeCla. The color is formed instantly if the ester is warmed for a short time on the H<sub>2</sub>O-bath, or allowed to remain for some time at the ordinary temp. These results indicate that the ester exists in an enolic and a ketonic form, both of which are liquids. The proportion of enolic form. which is in equilibrium is dependent largely on the solvents employed, it increases in the order MeOH, alc., Et<sub>2</sub>O and C<sub>2</sub>H<sub>4</sub>, the ratio deduced from the color intensity being 1.39, 1, 0.77 and 0.64, resp., i. e., for equal concs., the same color intensity is obtained with 0.64 pt. of a C.H. soln. as is given by 1 pt. of an alc. soln. Triethyl ammoniooxalosuccinate, C12HmO7N, from the ester and dry NH1, in Et1O; pale yellow crystals, softens 82°, m. 88-9°. At the ordinary temp., more quickly when warmed alone, or b. with alc., it is converted into triethyl α-aminoglutacone-β-carboxylate, H<sub>2</sub>NC(CO<sub>2</sub>Et): C(CO<sub>2</sub>Et)CH<sub>2</sub>CO<sub>2</sub>Et; colorless, lustrous plates from alc. or ligroin, m. 68-9°; b<sub>112</sub> 255-60°; b<sub>27</sub> 211-4°. It decolorizes Br-H<sub>2</sub>O immediately and quickly ppts. MnO, from KMnO<sub>4</sub>. The following salts were prepared, in aq. soln., from the above NH<sub>2</sub> deriv. Copper salt, (C<sub>18</sub>H<sub>17</sub>O<sub>7</sub>)<sub>2</sub>Cu, slender, green needles from Et<sub>2</sub>O + C<sub>8</sub>H<sub>8</sub>, m. 64-9°. Nickel sall, (C11H17O7)1Ni, slender, pale green needles from alc., m. 114-9°. Zinc sall, white needles from Et.O. These salts could not be prepared directly from the ester; the salts furnish a convenient means for the purification of the ester because it decomp, when distilled. Triethyl oxalosuccinate phenylhydrazone, PhNHN: C(CO,Et)CH(CO,Et)-CH<sub>2</sub>CO<sub>2</sub>Et, small, pale yellow plates from alc. + H<sub>2</sub>O, m. 85°. At 150°, or by the action of dry HCl on the phenylhydrazone, in Et<sub>2</sub>O, Et 1-phenyl-5-pyrazolone-3carbethoxy-4-acetate is formed, the corresponding acid being obtained from the phenylhydrazone and warm alc., NaOH. Ethyl 1-phenyl-5-acetoxypyrazole-3-carbethoxy-4acetate formula (I) below, is prepared from AcCl and the pyrazolone ester; colorless

$$\begin{array}{c} \text{PhN} & \text{C(OAc)} : \text{CCH}_{1}\text{CO}_{2}\text{H} \\ \text{N====}\text{CCO}_{2}\text{Et} \\ & \text{(I)} \end{array}$$

needles from alc., or ligroin, m. 89–90°. Aq., alkali hydroxides hydrolyze it to the above phenylpyrazolonecarboxyacetic acid, which, with AcCl, gives *r-phenyl-5-acetoxy-pyrazole-3-carboxy-4-acetic anhydride* (II); colorless, lustrous needles from C<sub>4</sub>H<sub>6</sub>, m. 150–1°. With alkalies it regenerates the parent acid, but b. alc. gives the *mono ester* of (I); colorless, lustrous needles from alc., or C<sub>6</sub>H<sub>6</sub>, m. 178–82°. It is sol. in aq. Na<sub>2</sub>CO<sub>2</sub>. In H<sub>2</sub>O, diphenylhydrazine hydrochloride and tri-Et K-oxalsuccinate give an additive compound, C<sub>24</sub>H<sub>20</sub>O<sub>7</sub>N<sub>2</sub>, of the ester and PhN<sub>2</sub>H<sub>3</sub>; crystals from Et<sub>2</sub>O, m. 81°. It passes slowly into triethyl oxalosuccinate diphenylhydrazone, C<sub>24</sub>H<sub>22</sub>O<sub>4</sub>N<sub>2</sub>; oil. In H<sub>2</sub>O, at the ordinary temp., HONH<sub>2</sub>.HCl, AcONa and the oxalosuccinic ester give a compound, C<sub>24</sub>H<sub>26</sub>O<sub>14</sub>N; slender, colorless needles from Et<sub>2</sub>O, softens 55°, m. 61–2°. As is known,

conc. HCl converts tri-Et oxalosuccinate into  $\alpha$ -ketoglutaric acid, HO<sub>2</sub>CCOCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>H, of which the following derivs. have been prepared. Diethyl ester, from the acid, alc. and HCl; colorless liquid with a slight odor, b<sub>22</sub> 160°. Phenylhydrazone, C<sub>11</sub>H<sub>12</sub>O<sub>4</sub>N<sub>2</sub>, obtained in dil. AcOH soln.; small tablets from AcOEt + CHCl<sub>2</sub>, m. 152-3°. In conc. H<sub>2</sub>SO<sub>4</sub>, with K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, or FeCl<sub>2</sub>, it gives an intense, purple-red color. At the ordinary temp., or more quickly when heated, the phenylhydrazone is converted into 1-phenyl-6-pyridazinone-3-carboxylic acid (III); which is best prepared, however,

$$\begin{array}{c} \text{PhN} & \begin{array}{c} \text{CO.CH}_3 & \\ \text{N:C(CO_3H)} \end{array} \\ \text{CH}_3 & \begin{array}{c} \text{C(CH_2CO_3Et)} \end{array} \end{array} \\ \text{CO.Et} \\ \text{O(III)} & \begin{array}{c} \text{CO.SEt} \end{array} \end{array}$$

by b. the ketobutaric acid with PhN<sub>2</sub>H<sub>2</sub> and glacial AcOH. Colorless plates from glacial AcOH, m. 172°. It gives no color with conc. H<sub>2</sub>SO<sub>4</sub> and K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, or FeCl<sub>3</sub>. When b. with alc. and dry HCl, (III) gives ethyl indole-α-carbethoxy-β-acetate (IV); colorless plates from alc., m. 83-4°.

J. BISHOP TINGLE.

Carbamide Derivatives of Phenylhydrazine. M. Busch and O. Limpach. Chem. Lab., Univ. Erlangen. Ber., 44, 1573-83.—α-Thiocarbanilido-β-carbanilidophenylhydrazine, PhNHCSNPhNHCONHPh, is obtained, in good yield, from Ph isocyanate and α-diphenylthiosemicarbazine, in C<sub>6</sub>H<sub>6</sub>; white needles from abs. alc., m. 164°. Its formation is accompanied by that of a compound free from S; slender, white needles, m. 214-5°. Yield, very poor. 1,4-Diphenyltriazolone-3-thiol (see below), is also produced simultaneously. These 3 substances are separated by soln. in alc. KOH (10%) from which H<sub>4</sub>O ppts. the second substance, AcOH the first and HCl the third. α-Carbanilido-β-thiocarbanilidophenylhydrazine, PhNHCONPhNHCSNHPh, is formed from Ph isocyanate and  $\beta$ -diphenylthiosemicarbazine, m. 178-9° not 170°. Warm, alc. KOH converts it into the above 1,4-diphenyltriazolone-3-thiol, m. 135°. a-Thiocarbanilido-β-carbanilido-o-tolylhydrazine, PhNHCSN(C,H,)NHCONHPh, from Ph isocyanate and 2-o-tolyl-4-phenylthiosemicarbazine; white needles, m. 181°. a-Thiocarbanilido-β-carbanilido-p-tolylhydrazine, from 2-p-tolyl-4-phenylthiosemicarbazine and Ph isocyanate; slender needles from alc., m. 174°. At 140°,  $\alpha$ - or  $\beta$ -diphenylthiosemicarbazine and Ph isothiocyanate give 1-phenyl-3-anilidothiobiazolone-5-anil, m. 156°. At the ordinary temp., a-diphenylthiosemicarbazine and Ph isothiocyanate fail to react; at 100° they give a mixture of  $\alpha$ - and  $\beta$ -thiosemicarbazine, while in b.  $C_aH_a$  the product is  $\beta$ -thiosemicarbazine, together with a little of the above bisazoloneanil. B.  $C_aH_a$  converts  $\alpha$ -diphenylthiosemicarbazine into a mixture of  $\alpha$ - and  $\beta$ -thiosemicarbazine, but the greater part of the a-diphenyl compd. is recovered unchanged. Et 2,4-diphenylthiosemicarbazine-1-carboxylate, PhNHCSN.PhNHCO,Et, is obtained from a-diphenylthiosemicarbazine and Et chlorocarbonate, in b. C.H.. It is identical with the compd. from PhNH, and Et chlorothiocarbonylphenylcarbazinate, CICSNPhNHCO<sub>2</sub>Et. β-Diphenylthiosemicarbazine and Et chlorocarbonate, when b. with CaHa, during about 5 hrs., form ethyl 1,4-diphenylthiosemicarbazine-1-carboxylate, PhNHCSNHNPhCO<sub>2</sub>Et; opaque leaflets from abs. alc., m. 175-6°. Yield, 75%. Alc. KOH converts it into 1,4-diphenyltriazolone-3-thiol. Et phenylcarbazinate and Ph isothiocyanate fail to react when mixed, or in presence of alc., or of CaHa, but with alc. KOH, at the ordinary temp., during 2 hrs., they give a mixture, which was separated as follows. With H<sub>2</sub>O a product (A) is deposited and from the filtrate H<sub>2</sub>SO<sub>4</sub> ppts. diphenyl-endoxytriazolthiol. From (A) Et,O extracts phenylthiourethane, PhNHCSOEt; the residue of (A) consists of the above Et 1,4-diphenylthiosemicarbazine-1-carboxylate. Yield, 33% of the original ester. This reaction involves a migration of the CO<sub>2</sub>Et from the  $\beta$ - to the  $\alpha$ -N at. of the hydrazine. In order to demonstrate this fact more clearly, Et sym.-phenylcarbazinate was treated with HNO, and the resulting yellow oil, which

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gave Liebermann's reaction, was converted by H<sub>2</sub>O into Et phenylazocarboxylate, C<sub>6</sub>H<sub>16</sub>O<sub>2</sub>N<sub>2</sub>; b<sub>2</sub> 130-2°. It is reduced without difficulty, by Zn dust, alc. and glacial AcOH, to Et phenylcarbazinate, m. 83°, which was identical with the parent ester in physical properties and in chemical reactions, as described above. *Hydrochloride*, from dry HCl, in Et<sub>2</sub>O; bundles of plates. It immediately regenerates the ester when treated with H<sub>3</sub>O. Et asym. phenylhydrazinoformate, H<sub>2</sub>NNPhCO<sub>2</sub>Et, was prepared from benzylidenephenylhydrazine and Et chlorocarbonate, in pyridine, the product being subsequently b. with dil., alc. H<sub>2</sub>SO<sub>4</sub>, to eliminate the benzylidene group. The ester has the properties described in the literature. It reacts very readily with Ph isothiocyanate, forming Et 1,4-diphenylthiosemicarbazine-1-carboxylate (see above).

I. BISHOP TINGLE.

Quaternary Ammonium Chlorides from Diphenylcarbamyl Chloride and Pyridine or Quinoline. J. HERZOG AND K. BUDY. Pharm. Inst., Univ. Berlin. Ber., 44, 1584-94.—Anhydrides may be prepared by the action of pyridine and aq., alkali hydroxides (equivalent to the halogen), on acid chlorides, at o°. In the case of diphenylcarbamyl chloride it is preferable to use its additive compd. with pyridine, diphenylcarbamylpyridinium chloride. When treated with NaOH, in the manner described, the following 2 compds. are formed. Asym. diphenylglutaconylcarbamide, Ph. NCON: CHCH: CHCH: CHOH, is separated by its more sparing sol. in MeOH; long, slender, very voluminous needles from C<sub>e</sub>H<sub>e</sub>, m. 189°. Its nature is shown by its conversion into diphenylcarbamide and glutaconic aldehyde, OCHCH: CHCH: CHOH, which was isolated as the dianiline deriv., PhN: CHCH: CHCH: CHNHPh. Diphenylcarbamic anhydride, (Ph.NCO), O, opaque, yellow needles from MeOH, m. 121-3°. Alc. NH2 converts it into asym.-diphenylcarbamide. Diphenylcarbamylquinolinium chloride, C22H1,ON2Cl, is prepared by the insolation, during about 3 wks., of a mixture of diphenylcarbamyl chloride and quinoline; red crystals from 10% HCl, m. 135-7°. Chloroplatinate, (C22H16ON2)2.H2PtCl6, yellow crystals from alc., m. 232°. Aq. NaOH, at oo, liberates from the chloride the hydroxide, C.H.: N(OH)CONPh. which transforms immediately in o C-hydroxy-N-diphenylcarbamyldihydroquinoline,

pale red, amorphous powder. B. alc. converts it into the *ethyl ether*, long, pale yellow needles, m. 195°. *Methyl ether*, from MeOH; yellow needles, m. 160-2°. J. B. T.

Hydroaromatic Compounds. A Hydrocarbon, C<sub>5</sub>H<sub>12</sub>, of the Semibenzene Series. K. AUWERS AND K. MÜLLER. Chem. Inst., Greifswald. Ber., 44, 1595-608.—1,1,4-Trimethyl-2,5-cyclohexadiene-4-ol formula (I) below, is prepared by treating the

$$Me_{3}C \stackrel{\text{CH}: CH}{\stackrel{\text{CH}: CH}{\stackrel{\text{CM}=CH}{\stackrel{\text{CH}: CH}{\stackrel{\text{CH}: CH}}{\stackrel{\text{CH}: CH}{\stackrel{\text{CH}: CH}}{\stackrel{\text{CH}: CH}{\stackrel{\text{CH}: CH}}{\stackrel{\text{CH}: CH}}}{\stackrel{\text{CH}: CH}}}{\stackrel{\text{CH}: CH}}{\stackrel{\text{CH}: CH}}}{\stackrel{\text{CH}:$$

ketone, O: C<sub>6</sub>H<sub>4</sub>MeCHCl<sub>2</sub> from p-cresol, CHCl<sub>3</sub> and aq. NaOH, with MeMgI and reducing the product (III) with Na and moist Et<sub>2</sub>O. It is purified by distillation;

colorless, lustrous needles, m. 43-4°; b<sub>13</sub> 60-100°; b. 169-70°. It has a penetrating odor and quickly volatilizes in air. At 0°, H<sub>2</sub>SO<sub>4</sub> (10%) converts (I) into 1,1-dimethyl-4-methylene-2,5-cyclohexadiene (II); colorless, mobil oil with a sweet, pleasing odor,

**b**<sub>m</sub> below 50° (in a current of H);  $d_4^{15.85}$  0.8687;  $n_a^t$  1.50363;  $n_D^t$  1.50850;  $n_B^t$  1.52136;  $\mathbf{m}_{\tau}^{t}$  1.53292 (t 15.45°);  $\mathbf{M}_{\alpha}$  40.90;  $\mathbf{M}_{D}$  41.24;  $\mathbf{M}_{\beta}$ - $\mathbf{M}_{\alpha}$  1.21;  $\mathbf{M}_{\tau}$ - $\mathbf{M}_{\alpha}$  1.99. These values refer to material which had not been heated. After 3 distillations, in H, the following data were obtained:  $d_4^{16-2}$  0.8430;  $n_a^t$  1.50309;  $n_D^t$  1.50861;  $n_B^t$  1.52298;  $n_\tau^t$  1.53610 (t 14.95°);  $M_{\alpha}$  42.11;  $M_{D}$  42.50;  $M_{\beta}$ - $M_{\alpha}$  1.40;  $M_{\gamma}$ - $M_{\alpha}$  2.31; EM 2.23, 2.34, 0.49 and 0.85, resp.; E $\Sigma$  1.86, 1.95, 54 % and 58%, resp. For the sake of comparison similar determinations have been made in the case of pseudocumene, mesitylene and p-methylethylbenzene, all of which were distilled immediately before the observations were made. Pseudocumene,  $d_4^{14-1}$  0.8805;  $n_{\alpha}$  1.50316;  $n_{D}$  1.50780;  $n_{\beta}$  1.51909;  $n_{\gamma}$  1.52878, at 13.9°; Ma 40.32; MD 40.61; Mg-Ma 1.07; Mr-Ma 1.72; EM, 0.44, 0.48, 0.16 and 0.26, resp.;  $\mathbf{E}\Sigma$ , 0.37, 0.40, 18% and 18%, resp. Mesitylene,  $\mathbf{d}_{4}^{14.4}$  0.8697;  $n_{\alpha}$  1.49774;  $m_D$  1.50228;  $n_B$  1.51333;  $n_T$  1.52311, at 14.2°;  $M_\alpha$  40.45;  $M_D$  40.76;  $M_{\beta}-M_{\alpha}$  1.07;  $M_7-M_a$  1.73; EM, 0.57, 0.60, 0.16 and 0.27, resp.; E $\Sigma$ , 0.47, 0.50; 18% and 18%, resp. p-Methylethylbenzene, d<sub>4</sub><sup>15.8</sup> 0.8664; n<sub>a</sub> 1.49325; n<sub>D</sub> 1.49750; n<sub>B</sub> 1.50831; n<sub>r</sub> 1.51779, at 15.2°; Ma 40.28; MD 40.57; Mg-Ma 1.04; My-Ma 1.69; EM, 0.40, 0.41, 0.13 and 0.23, resp.;  $E\Sigma$ , 0.33, 0.34; 14% and 16%, resp. The pseudocumene, from (II) during its purification, had  $d_4^{15.8}$  0.8824;  $n_{\alpha}$  1.50252;  $n_{D}$  1.50702;  $n_{\beta}$  1.51825;  $n_{\gamma}$  1.52818, at 14.8°;  $M_{\alpha}$  40.17;  $M_{D}$  40.47;  $M_{\beta}$ – $M_{\alpha}$  1.05;  $M_{\gamma}$ – $M_{\alpha}$  1.71. When protected from air (II) changes only slowly, otherwise it passes quickly into a viscid material. The reduction carried out during the preparation of (I) is not complete and a portion of the 1,4-dimethyl-1-dichloromethyl-2,5-cyclohexadiene-4-ol (III), is recovered; b,4 126-7°; m. indefinitely. It consists of a mixture of stereoisomers, the ratio of which is not changed by b.; from petroleum ether the higher m. modification is deposited, m. 96°. Portions of (III) which have been prepared directly, without treatment with Na, are much less stable. They decomp. when heated, H,O being eliminated, and they liquefy after being retained, at the ordinary temp., during about 14 dys. This applies to material which has been recrystallized several times. The recovered substance undergoes no apparent change after 3 mos. At oo, conc. H,SO, converts (III) into 2,4-dimethylbenzaldehyde. J. BISHOP TINGLE.

Ketenes. XIX. Formation and Preparation of Diphenyl Ketene. H. STAUD-INGER. Chem. Inst., Techn. Hochsch. Karlsruhe. Ber., 44, 1619-23; see C. A., 5, 1768.—Diphenyl ketene is formed when well cooled Pr. aN is added to diphenylacetyl chloride, both in abs. Et,O, in an atm. of H. Yield, almost quant. Quinoline, or quinaldine act much less completely than Pr, aN. Some diphenyl ketene is also produced when diphenylacetyl chloride is heated, but the yield is poor. The other products are HCl, CO, diphenylchloromethane and tetraphenylethylene. The best results are obtained by distilling the chloride, under the ordinary pressure, in a current of CO. Yield, about 14% of the chloride. Diphenylacetyl chloride, b<sub>15</sub> 178°. The best method of preparing diphenyl ketene is the following: hydrazibenzil is oxidized by yellow HgO to azibenzil; the crude C<sub>a</sub>H<sub>a</sub> soln. of this is filtered, dried by means of CaCl, and then slowly run into a Claissen distillation flask, which is heated at 100° and through which a current of dry CO<sub>2</sub> is passing. Yield, 64%, which is also stated to be about 70%. In the flask there remains a compound, C28H20O2N2; yellow crystals from glacial AcOH, m. 201°. Sometimes a quantity of tetraphenyldiketocyclobutane (dimoldiphenyl ketene) is also produced. See following abstr. J. BISHOP TINGLE.

Ketenes. XX. Attempts to Prepare Quinoketenes. H. STAUDINGER AND K. CLAR. Chem. Inst. Techn. Hochsch., Karlsruhe. Ber., 44, 1623-33; see preceding abstr.—The object of the expts. described below was the prep. of "quinoketene,"  $C_0H_4(:CO)_2$ ; in this respect they were unsuccessful. The halogen from terephthalyl chloride, or bromide could not be removed by the action of Zn, or of Ag, in Et<sub>2</sub>O, or AcOEt. p,p-Dichlorocarboxydiphenyldichloromethane, Cl<sub>2</sub>C(C<sub>2</sub>H<sub>4</sub>COCl)<sub>2</sub>, is prepared

from the acid, or from benzophenone-p,p-dicarboxyl chloride and PCla, at 140°; colorless crystals from CS<sub>2</sub> + petroleum ether, m. 78°. Yield, about 80%. It is relatively stable towards H<sub>2</sub>O and is not changed by treatment with Zn, or with Ag. Terephthalyl bromide, CaH4(COBr), from the acid, PBr, and POBr, long, colorless needles petroleum ether, m. 85°. Yield, 70%. **♦-Carboxytriphenylcarbinol.** HOCPh,C,H,CO,H, is best prepared from diphenyl-p-tolylcarbinol, MeC,H,CPh,OH (50 g.), conc. HNO<sub>2</sub> (100 cc.) and H<sub>2</sub>O (400 cc.), which are b. during 40 hrs. Yield, 88%. With PCla or preferably SOCla the acid gives p-chlorocarboxyltriphenylchloromethane, CICPh,C,H,COCI; colorless crystals from petroleum ether, m. 80-1°. It is very stable towards H<sub>2</sub>O and with PhNH, forms p-anilidocarboxytriphenylcarbinol; crystals from glacial AcOH, m. 182°. p-Carboxytriphenylmethane, is best prepared from the above carbinol acid, I, P and glacial AcOH, which are b. during 40 hrs. Yield, 84%. Chloride, from the acid and SOCl,; white crystals from petroleum ether, m. 88-90°. It is relatively stable towards H<sub>2</sub>O and is not changed by Pr<sub>2</sub>aN, in abs. Et<sub>2</sub>O + petroleum ether; with PhNH, it forms the anilide, C<sub>22</sub>H<sub>21</sub>ON; crystals from glacial AcOH, m. 196°. When shaken at the ordinary temp., during 3 days, in an atm. of CO2, with Hg and abs. Et.O, p-chlorocarboxytriphenylchloromethane is converted into p-chlorocarboxytriphenylmethyl, Ph<sub>2</sub>CC<sub>2</sub>H<sub>4</sub>COCl, which was obtained only in soln.; deep violet. It is oxidized by air to the peroxide, Can Han O. Cla; white scales from dichloroethylene + petroleum ether, m. 168°. Yield, 78%. It gives the usual reactions of the peroxides and is converted by conc. H<sub>2</sub>SO<sub>4</sub> into p-carboxytriphenylcarbinol. p-Carbomethoxytriphenylcarbinol, CnH12O2, from the Ag salt and MeI; crystals from MeOH, m. 119°. With PCl, it gives p-carbomethoxytriphenylchloromethane, oil. This, when shaken with Hg, in the manner described above forms p-carbomethoxytriphenylmethyl; reddish violet. It was obtained only in soln, and is stable towards H<sub>2</sub>O and PhNH<sub>2</sub>. Air converts it into the *peroxide*; white powder from dichloroethylene + petroleum ether, m. 171.5° (decomp.). It gives the ordinary peroxide reactions. See following abstrs. J. BISHOP TINGLE.

Ketenes. XXI. Attempts to Prepare Allene Ketene. H. STAUDINGER AND E. OTT. Chem. Inst. Techn. Hochsch., Karlsruhe. Ber., 44, 1633-7; see preceding abstrs.—α-Bromocinnamyl chloride, PhCH: CBrCOCl, is best prepared from SOCl, instead of PCla. a-Bromocinnamyl bromide, PhCH: CBrCOBr, is obtained from the acid and PBr, in C<sub>e</sub>H<sub>s</sub>, at the b. p.; colorless liquid, b<sub>m</sub> 170°. Vield, 67%. Only resinous products containing halogen could be isolated by the interaction of the above chloride or bromide and "Zn wool," in AcOEt, in an atm. of CO. Dimethylacrylic ester is best prepared from bromoisovaleric ester and quinoline, at 140-4°, during 15-20 hrs. Yield, 75-80%. It was hydrolyzed and the resulting acid brominated, thus giving dibromoisovaleric acid, which, with EtONa, forms  $\alpha$ -bromo- $\beta$ ,  $\beta$ -dimethylacrylic acid, Me<sub>2</sub>C: CBrCO<sub>2</sub>H. Yield, quant. Chloride, from the acid and SOCl<sub>2</sub>; colorless liquid, b. 178°; b<sub>16</sub> 73°. Anilide, crystals from petroleum ether, m. 90°. With "Zn wool," in the manner described above, the chloride gives only a resin. Dimethylacrylyl chloride, from the acid and SOCl<sub>2</sub>; b. 145-50°. Anilide, C<sub>11</sub>H<sub>12</sub>ON, crystals from petroleum ether, m. 126-7°. Benzalmalonyl chloride, PhCH: C(COCI), is prepared from the acid and PCl, in b. petroleum ether, and is purified by washing with H<sub>2</sub>O, at o°; white needles from petroleum ether, m. 37°. Yield, 70%. It could not be converted into benzalmalonyl anhydride. It forms a resin when b. with Ago and Et<sub>2</sub>O, or C<sub>8</sub>H<sub>8</sub>. In petroleum ether, with pyridine, an additive compound, C<sub>20</sub>H<sub>16</sub>O<sub>2</sub>N<sub>2</sub>Cl<sub>2</sub>, is produced; pale yellow, voluminous powder, decomp. 95-100°. See following abstr. I. BISHOP TINGLE.

Ketenes. XXII. Attempts to Prepare Phenyl Methoxy Ketene. H. STAUD-INGER AND O. KUPFER. Chem. Inst. Techn. Hochsch., Karlsruhe. Ber., 44, 1638-40;

see preceding abstrs.—Phenylmethoxyacetyl chloride, MeOCHPhCOCl, is prepared from the acid and SOCl<sub>2</sub>; colorless liquid, b<sub>0·1</sub> 80-1°. It decomp. at 120°, or when distilled (15 mm.) into CO, BzH and MeCl and it does not react with Pr<sub>2</sub>"N, in petroleum ether, nor with Br, at the ordinary temp. When the soln. is warmed decomp. takes place in the manner stated above.

J. BISHOP TINGLE.

Anthraquinone Series. IV. Anthraquinone-2,1-acridone and Anthraquinone-diacridone. FRITZ ULLMANN AND PAUL OCHSNER. Techn. Chem. Inst. Roy. Techn. Hochsch., Berlin. Ann., 381, 1-11.—a-Chloroanthraquinone formula (I) below, is

best prepared by dissolving K anthraquinone-a-sulfonate (40 g.) in crude, conc. HCl (170 cc.) and H<sub>2</sub>O (1200 cc.) and adding slowly, during 3 hrs., to the b. liquid, NaClO<sub>2</sub> (40 g.), in H<sub>2</sub>O (200 cc.). The mixture is b. during 1 hr. longer and the product, after being washed, is quite pure. Yield, 95%. This method may be used with advantage for the identification and evaluation of anthraquinonesulfonic acids. Anthraquinone*s-amilino-o-carboxylic acid* (II), is obtained either from (I) and anthranilic acid, or from o-chlorobenzoic acid and α-aminoanthraquinone, in both cases in presence of "natur" Cu; Bordeaux-red scales from glacial AcOH, m. 282° (cor.). Yield, 99 and 48%, resp. In pyridine the color is red, changing to violet with H<sub>2</sub>O; in conc. H<sub>2</sub>SO<sub>4</sub> green, violet flocks being pptd. on dilution. Alkali salts, bluish violet and very sparingly sol, in H<sub>2</sub>O. At 100-10°, during 30 mins., conc. H<sub>2</sub>SO<sub>4</sub> converts (II) into anthraquinone-2,1-acridone (III); violet lustrous, interlaced needles from PhNH2, or PhNO2, m. 380°. With NaOH and Na hyposulfite it gives a bluish violet vat-dye, which imparts the same shade to cotton; on exposure to air this color changes to orange-red, then to bluish red. Yield, 94%. In b. xylene, PCl, and (II) form anthraquinone-tanilino-o-carboxyl chloride, Bordeaux-red needles; when warmed with PhNO, it is converted into (III). Yield, 76%. The product is purer than that obtained by the use of H<sub>2</sub>SO<sub>4</sub>, because no sulfonic acid is formed. Anthraquinonebromoacridone, C<sub>m</sub>H<sub>10</sub>O<sub>2</sub>NBr, is obtained from (III) and Br, in PhNO<sub>2</sub>; red, stellate needles, m. 339° (cor.). In PhNO, or pyridine the color is wine-red; in conc. H<sub>2</sub>SO<sub>4</sub>, 1ed with a slight yellow tinge. Yield, 104% of (III). Anthraquinone-1,5-dianilino-0,0-dicarboxylic acid, (IV), is prepared at 200°, from 1,5-dichloroanthraquinone, anthranilic acid, fused AcOK, and PhNO, together with a little Cu(OAc), and "natur" Cu; reddish violet needles from glacial AcOH, m. 349° (cor.). In pyridine the color is reddish violet, changing to bluish violet on adding H<sub>2</sub>O. Yield, 85% of the dichloroanthraquinone. At 100-10°, conc. H<sub>2</sub>SO<sub>4</sub> converts (IV) into anthraquinone-2,1,6,5-diacridone (V);

bluish violet needles with a metallic luster from PhNO<sub>2</sub>, m. above 360°. At the b. p. it dissolves in 1000 pts. of PhNO<sub>2</sub>; in pyridine the color is bluish violet. Yield, 90% of (IV). The vat dye, prepared in the manner described above, is blue and it imparts bluish violet shades to cotton. In presence of naphthalene, PCl<sub>2</sub> converts (IV) into

the carboxyl chloride which, when heated, gives (V), in the manner described above.

J. BISHOP TINGLE.

Action of Iodine on Benzoic and Salicylic Acids. N. Bungs. Lab., Univ. Kiev. J. Russ. Phys.-Chem. Soc., 41, 455-60; thru Chem. Zentr., 1909, II, 814.—The compound, Bz<sub>2</sub>O.AgIO, is prepared from I and BzOAg, with or without C<sub>0</sub>H<sub>6</sub> as solvent; small prisms from C<sub>0</sub>H<sub>6</sub>. It is blackened by NH<sub>2</sub> and is decomp. into BzOH by the action of moisture. No corresponding deriv. of salicylic acid could be obtained.

J. BISHOP TINGLE.

Synthesis of Methoxycinnamic Acid. N. Bunge. Lab., Univ. Kiev. J. Russ. Phys.-Chem., Soc., 41, 460-3; thru Chem. Zentr., 1909, II, 819.—Ethyl methoxycinnamate is prepared by well cooling and mixing Et bromoacetate (67 g.), anisic aldehyde (48 g.) and Zn. After a week the product is treated in the usual manner and the ester obtained from the portion b<sub>28-21</sub> 195-202.5°. Plates from alc., m. 49-50°. Yield, 34.9 g. Bromo derivative, m. 111.5°. Methoxycinnamic acid, from the ester; m. 170-1°. Silver salt, C<sub>10</sub>H<sub>9</sub>O<sub>2</sub>Ag.

J. BISHOP TINGLE.

Reduction of Nitrobenzene to Aniline. C. NICOLESCU OTIN. Electrochem. Lab. Techn. Hochesh., Charlottenberg. Z. Elektrochem., 16, 674-80; thru Chem. Zentr., 1910, II, 1043.—A study of the results hitherto obtained from expts. on the electrolytic reduction of PhNO<sub>2</sub> to PhNH<sub>2</sub>, leads to the conclusion that, in order to render the process available for technical use, the cathode surface must be large and the HCl conc. must be confined within definit limits. The author has employed aq. HCl (d. 1.19) and has filled the cathode compartment with bulky material, such as "lead wool" or Ni wire, which is in contact with the cathode liquid. Under these conditions, with a 3-4 volt current, the yield of PhNH<sub>2</sub> was 90-5%, calculated on the electricity. Considerable quantities of chloroanilines are produced with HCl of other cones.

J. BISHOP TINGLE.

Action of Nitric Acid on Saturated Hydrocarbons. IV. S. Nametein. J. Russ. Phys.-Chem. Soc., 42, 581-5; through Chem. Zentr., 1910, II, 1376.—The yield of nitro products is increased by the use of Al(NO<sub>2</sub>)<sub>2</sub>. Hexamethylene (78 g.) and Al(NO<sub>2</sub>)<sub>3</sub> (240 g.) give 56.7% of mononitro compd., together with what is probably ketohexamethylene, C<sub>2</sub>H<sub>10</sub>O, and dinitrodicyclohexyl, C<sub>12</sub>H<sub>20</sub>(NO<sub>2</sub>)<sub>2</sub>; m. 216.5-7°. This compd. is also obtained by the action of mol. Ag on 1,1-bromonitrohexamethylene, C<sub>2</sub>H<sub>10</sub>BrNO<sub>2</sub>; b<sub>20</sub> 116°; d<sub>4</sub><sup>10</sup> 1.5148.

J. BISHOP TINGLE.

Action of Nitric Acid on Methylcyclohexane. S. NAMETKIN. Moscow. J. Russ. Phys.-Chem. Soc., 42, 691-701; thru Chem. Zentr., 1910, II, 1377.—The yield of nitro compds. from methylcyclohexane is 58% by the use of HNO, (d. 1.2); with Al(NO<sub>2</sub>), the yield is 72%. The primary and secondary nitro compds. are separated by means of their sol. in aq. KOH. 1,1-Nitromethylcyclohexane, C,H13NO, liquid  $b_{40}$  109-10°;  $d_4^0$  1.0547;  $d_4^{20}$  1.0384;  $n_{20}$  1.4580. It is oxidized by HNO<sub>2</sub> to succinic acid and a little oxalic acid. 1,1-Aminomethylcyclohexane, C,H13NH2, from the preceding compd., Sn and HCl; oil,  $b_{744}$  143°;  $d_4^0$  0.8729;  $d_4^{19}$  0.8568;  $n_{19}$  1.4536. Benzoyl derivative, needles from dil. alc., m. 100.5-1°. 1,3-Nitromethylcyclohexane, is formed together with the above isomer and is separated by its smaller sol. in dil., aq. NaOH; liquid, b40 119-20°; d4 1.0547; d49 1.0382; n19 1.4618. 1,3-Aminomethylcyclohexane, from the nitro compd.; liquid b<sub>752</sub> 152-3°; d<sub>4</sub><sup>19</sup> 0.8562; n<sub>10</sub> 1.4558. Benzoyl derivative, m. 95-7°. Oxidation converts the amine into a mixture of  $\alpha$ - and  $\beta$ -methyladipic acids and  $\beta$ -methylcyclohexanone, C<sub>7</sub>H<sub>12</sub>O; liquid, b. 168-9°; d<sub>4</sub><sup>17</sup> 0.9179;  $n_1$ , 1.4453. Semicarbazone, m. 179-80°. Hexahydrophenylnitromethane, is also formed, together with the nitration products described above; liquid, b<sub>40</sub> 123-4°; d<sub>4</sub><sup>18</sup> 1.0459; n<sub>18</sub> 1.4684. The oxidation of methylcyclohexane, by means of HNO<sub>1</sub>, gives rise to a mixture of adipic, succinic, oxalic, glutaric and pyrotartaric acids. Nitration of the naphtha fraction, b. 100–2°, gave results very similar to those described above. The differences noted were all referable to the fact that the parent substance from naphtha was not completely homogeneous.

J. BISHOP TINGLE.

Cyclohexylpseudonitrol. S. Nametkin. Moscow. J. Russ. Phys.-Chem. Ges., 42, 585-6; thru Chem. Zentr., 1910, II, 1377.—Cyclohexylpseudonitrol,  $C_0H_{10}O_2N_3$ , is prepared by adding NaNO<sub>3</sub> and dil.  $H_2SO_4$  to nitrohexamethylene, in aq. KOH, at 0°. A blue oil is first deposited, which soon changes into colorless crystals, m. 70-1°. The oil and the crystals are supposed to be the mono- and the dimol. form, resp., of the nitrol. In AcOH, CrO<sub>3</sub> oxidizes the nitrol to 1,1-dinitrohexamethylene,  $C_0H_{10}(NO_3)_3$ ; yellow oil  $b_{25}$  142-3°;  $d_2^{21}$  1.2452;  $n_{21}$  1.4732.

J. BISHOP TINGLE.

Electrochemical Reduction of Organic Halogen Compounds. K. Brand. Z. Elektrochem., 16, 669-72; thru Chem. Zentr., 1910, II, 1471. - Diphenyldichloroethane, Cl<sub>2</sub>CHCHPh<sub>2</sub>, is the chief product of the electrolytic reduction of asym.-diphenyltrichloroethane. A little diphenylchloroethylene, ClCH: CPh, is also obtained; it is formed by the action of NaOH on the above dichloro compd. The electrolysis was conducted by means of a Pb cathode, in an alc. soln. containing AcONa and diphenyltrichloroethane. The anode liquid contained Na<sub>2</sub>CO<sub>2</sub>. Similar results were obtained in alc. HCl, even when 4 times the calculated current was employed. Evolution of H begins in the alk. soln. only when reduction to the dichloro compd. has taken place; in the acid soln. it begins much earlier. At the temp, of a b, H,O bath, in presence of alc. HCl, the products are diphenyldichloroethane, a little diphenylchloroethylene and sym.-tetraphenyldimethylacetylene (tetraphenylcrotonylene). Ph<sub>2</sub>CHCCC: HPh<sub>2</sub>; m. 114-5°. In b. amyl alc., Na reduces it to tetraphenylbutane and it is oxidized to diphenylacetic acid by KMnO4 and MgSO4, in acetone. Ditolyltrichloroethane, when reduced electrolytically, in the manner described above, gives \$.\$'-dimethylstilbene and a hydrocarbon. J. BISHOP TINGLE.

Ketenes. XXIII. Reactivity of Halogen Atoms towards Metal. H. STAUDINGER. K. CLAR AND E. CZAKO. Chem. Inst., Techn. Hochsch., Karlsruhe. Ber., 44, 1640-7; see C. A., 5, 3244.—The following 4 series of compds. have been tested with regard to their reactivity with Zn, in presence of Et.O: (1) MeI, CH.I., CHCl., CCl., (2) Benzyl chloride, PhCH<sub>2</sub>Cl, benzal chloride, PhCHCl<sub>2</sub>, and phenyltrichloromethane, PhCCl<sub>2</sub>. (3) Diphenylchloromethane, Ph,CHCl and diphenyldichloromethane. Ph<sub>2</sub>CCl<sub>2</sub>. (4) Triphenylchloromethane, Ph<sub>2</sub>CCl. The reactivity appears to be influenced by the presence in the mol. of Ph and Cl, the former having the greater effect. because, of the compds. mentioned above, only PhCCl, Ph,CCl, and Ph,CCl react with Zn. "Dimethoxydiphenyldichloromethane" (MeOC,H4),CCl2, and Zn give "tetramethoxytetraphenylethylene," but "dicarbomethoxydiphenyldichloromethane." (MeO<sub>2</sub>CC<sub>2</sub>H<sub>4</sub>)<sub>2</sub>CCl<sub>2</sub>, is without action. Diphenyldichloromethane and Zn form tetraphenyldichloroethane, together with some tetraphenylethylene. This latter is obtained alone if the time of heating be prolonged. "Dicarbomethoxydiphenyldichloromethane" is prepared from "di-Me benzophenonedicarboxylate" and PCl<sub>3</sub>, at 150°; colorless needles from petroleum ether, m. 73°. It is very stable. Dichlorostilbene is produced from phenyltrichloromethane and Zn. Isobutylphenylchloroimide, Me<sub>2</sub>CHCCl: NPh, is obtained from isobutyric anilide and PCl<sub>3</sub>, at 100°; colorless liquid, b., 102-4°. Diphenylacetylphenylchloroimide, Ph.CHCC1: NPh, is prepared from the anilide and PCl<sub>s</sub>; colorless crystals from abs. Et<sub>2</sub>O, m. 90°. It is easily decomp. by H.O. Triphenylacetylphenylchloroimide, Ph.CCCl: NPh, is prepared like the preceding compds.; colorless, opaque crystals from ligroin, m. 132°. These chloroimides fail to react with Mg, in AcOEt. Which of the isomeric methoxy and carbomethoxy compds. were employed, is not stated. Speculations are given regarding the mode of reaction of carboxyl chlorides.

J. BISHOP TINGLE.

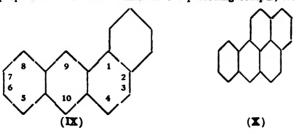
Vat Dyes from α-Naphthoquinone. R. PUMMERER AND K. BRASS. Chem. Techn. Lab., Techn. Hochsch., Munich. Ber., 44, 1647-56.-2-[p-Aminoanilino]-1,4-naphthoquinone, (O:) C10H4NHC4H4NH2, is prepared by reducing the nitro compd., by means of alkaline hyposulfite; black-violet prisms from xylene, m. about 214°. In alc. the color is violet-red; in CHCl, red; in conc. H,SO, safranine red. Sulfate, yellowish red. N,N'-Bis-[1,4-naphthoquinonyl-2]-p-phenylenediamine, [(O:), C10H4NH hC4H4, is obtained by b. a-naphthoquinone with p-phenylenediamine and glacial AcOH; brownish violet, granular, cryst. powder, m. about 250°. Its vat dye colors cotton brownish violet, changing to violet in conc. HCl. The color is "wash-fast." The amine itself gives a violet color with conc. H<sub>2</sub>SO<sub>4</sub> and a yellowish red with PbO<sub>2</sub>, xylene and AcOH. 2-[Diphenylene-4'-amino]-1,4-naphthoquinone, (O:),C10HaNHCaHaPh, is formed from α-naphthoquinone and 4-aminodiphenyl, in alc.; red needles, m. 215°. In conc. H.SO. the color is reddish violet. Its vat-dye is orange, it dyes cotton scarlet, the color being fairly "wash-fast." 2-Bensidino-1,4-naphthoquinone, (O:),C10H2NH-C.H.C.H.NH, prepared like the preceding compd., from benzidine; brown needles from PhNO, m. 259°. In organic solvents the color is blood red, in conc. H.SO. reddish violet. Yield, 66%. Acetyl derivative, C, H, O, N, from AcOH and Ac,O; violet crystals, m. 329° (decomp.). In conc. H2SO4 the color is dark violet. The compd. dyes cotton bluish red, the color being "wash-fast." 2-o-Tolidino-1,4-naphthoquinone, CaHaO2N2, is prepared in glacial AcOH, from o-tolidine; dark violet powder. Its vat dye colors cotton a fast reddish brown shade. Acetyl derivative, C. H., O. N., ruby-red, spindle-shaped prisms from toluene, m. 233°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is reddish violet. The compd. dyes cotton a rose color, which is fairly fast to washing. 2-[o-Dichlorobenzidino]-1,4-naphthoquinone, C. H14O2N2Cl2, from o-dichlorobenzidine, in glacial AcOH; m. 237°, previously softening. In neutral, organic solvents the color is yellowish red, in glacial AcOH, bluish red, in conc. H<sub>2</sub>SO<sub>4</sub>, cherry-red. Yield, good. Acetyl derivative, CatH10O2N2Cl2, large, transparent, light brown, microscopic plates with a coppery luster, m. 264°. Benzoyl derivative, from BzCl, in b. PhNO,; small transparent, microscopic, prismatic plates with a coppery luster, m. 302°. The amine dyes cotton bluish red, the acetyl deriv. yellowish red, the colors being very fast to washing. \_1,4-Naphthoquinonyl-2-p,p'-diaminodiphenylcarbamide, (O:)<sub>2</sub>C<sub>10</sub>H<sub>2</sub>NHC<sub>2</sub>H<sub>4</sub>NHCONH- $C_nH_nNH_n$  from sym.  $p_np'$ -diaminophenylcarbamide, in glacial AcOH; dark red, decomp. 348°. In alc. the color is reddish violet, in conc. H<sub>2</sub>SO<sub>4</sub> cherry-red. It dyes cotton reddish violet, the color being "wash-fast." 1,4-Naphthoquinonyl-2-p,p'-diaminobenzophenone, (O:)2C10H2NHC0H4COC4H4NH2, from p,p'-diaminobenzophenone, in glacial AcOH; flat, brick-red prisms from alc. The vat dye is reddish yellow, it colors cotton orange, the shade being "wash-fast." 1,4-Naphthoquinonyl-2-p,p'-diaminodiphenylmethane, (O:)2C10H4NHC4H4CH2C4H4NH2, from p,p'-diaminodiphenylmethane, in alc.; red crystals from xylene, m. 182°. It dyes cotton light red, the color being only medium "wash-fast." The authors record some expts. made, in an atm. of CO. to test the stability, when washed, of the colors obtained on vegetable fiber, presumably cotton, by means of various leuco dyes. The washing liquid contained 5 g. of soap per 1., the temp. 70-80° (time not stated); leucoflavanthrene is extremely fast, "tetrabromoindigo white" less so, whereas leucoindigo and leucothioindigo are very fugitive. This shows that, as would natuarally be expected, the mutual affinity of the fiber and the leuco compd. is different from that of the fiber and the dye.' J. BISHOP TINGLE.

Action of Glycerol and Sulfuric Acid on Amino Compounds and on Substances Free from Nitrogen in the Anthracene Series: Benzanthrone and its Reduction Products: Remarks on the Nomenclature and Designation of the Position in Complex Cycloids of the Anthracene Series. OSCAR BALLY AND ROLAND SCHOLL. Ludwigshafen a/R and Graz. Ber., 44, 1656-70.—In addition to the compd., C<sub>20</sub>H<sub>11</sub>ON, which is produced from 2-, or 1-aminoanthraquinone, glycerol and conc. H<sub>2</sub>SO<sub>4</sub> (Ber., 38, 194 (1905)), there is also formed 2,3-pyridinoanthraquinone formula (I) below, which is separated

by its greater solubility in dil. H<sub>2</sub>SO<sub>4</sub>; yellow needles from PhNH<sub>2</sub> m. 322°. At 120°, anthranol (1pt.), glycerol (1pt.) and H<sub>2</sub>SO<sub>4</sub> (15 pts.; 82%) give benzanthrone (II), which is also prepared by adding glycerol and H<sub>2</sub>SO<sub>4</sub> (82%) to a mixt. of anthraquinone H<sub>2</sub>SO<sub>4</sub> (82%) and (PhNH<sub>2</sub>)<sub>2</sub>.H<sub>2</sub>SO<sub>4</sub>, at 100°. Zn dust and aq. NaOH, or NH<sub>2</sub>, or alkaline hyposulfite soln. reduces (II) to dihydrobenzanthrone (III), which was not purified completely. When (II) is distilled over glowing pumice and Zn dust, in a current of H, it is converted into benzanthrene (IV), or (V) which is best purified by

$$(V) \qquad (VI) \qquad (VII) \qquad (VIII) \qquad (VIII)$$

means of its picrate; small, highly lustrous, pale yellow plates from alc. (in an atm. of CO<sub>2</sub>), m. 84°. Its solns, have a green fluorescence. In conc. H<sub>2</sub>SO<sub>4</sub> the color is red with a reddish brown fluorescence; the soln, evolves SO<sub>3</sub>. In air (IV) is oxidized to (II). Picrate, C<sub>17</sub>H<sub>12</sub>,C<sub>2</sub>H<sub>2</sub>(NO<sub>2</sub>)<sub>2</sub>OH, slender, dark red needles from alc., m. 110-1°. 10-Dibromobenzanthrene (VI), is prepared from (IV) and Br. in glacial AcOH; yellow crystals from alc., m. 174°. In conc. H<sub>2</sub>SO<sub>4</sub> it exhibits a red to reddish brown fluorescence. Dihydrobenzanthrene (VII), or (VIII), is obtained from (II), HI (b. p. 127°) and red P, by b. during 10 hrs.; yellow, concentric needles from alc., m. 81°. In conc. H<sub>2</sub>SO<sub>4</sub> its fluorescence is red to reddish brown, the soln, evolves SO<sub>2</sub>. Picrate, C<sub>17</sub>H<sub>14</sub>,C<sub>2</sub>H<sub>2</sub>(NO<sub>2</sub>)<sub>2</sub>OH, feathery aggregates of orange-yellow, lustrous needles from alc., m. 125°. Bromodihydrobenzanthrene, C<sub>17</sub>H<sub>12</sub>Br, from (VII) and Br (2 mols.), in glacial AcOH; colorless, lustrous crystals from dil. AcOH, m. 123°. Its fluorescence in warm, conc., H<sub>2</sub>SO<sub>4</sub> is reddish brown to brown. Dibromodihydrobenzanthrene, C<sub>17</sub>H<sub>12</sub>Br<sub>2</sub>, is prepared in a similar manner to the preceding compd., from 4 mols. of



Br; needles with a yellow shade from glacial AcOH, m. 157°. The authors suggest a system of nomenclature for derivs. of anthracene and anthraquinone based on the following principles: (1) The polynuclear compds. are regarded as derivs. of anthracene, the C ats. of which are numbered in the usual manner. The additional nuclei are designated by their specific names, preceded by the numbers of the anthracene C ats. to which they are linked. Thus (IX) and (X) are termed 1,2-benzanthracene (Graebe's naphthanthracene) and 2,9-naphthanthrone, resp. (2) When 2 or more independent nuclei are linked to the anthracene, the name begins with nucleus of simplest comp. and of most intimate linkage. [The authors fail to state what is to be done if the simplest nucleus is not that which is most intimately linked. Moreover, in their examples, they follow exactly the reverse of the plan they suggest, of the isocyclic nuclei being named before the heterocycloids. Abstr.]. The compds. (XI)

$$(\mathbf{XI}) \qquad (\mathbf{XII}) \qquad (\mathbf{XIII})$$

and (XII) are termed 3,4,1,9-dibenzanthrone and 3,4-pyridino-1,2-benzanthraquinone, resp. (3) Substituents in the nuclei which are linked to the anthracene are indicated by numbers which are specific for each nucleus. The numbers begin at the end which is nearest to the C at. of lowest number in the anthracene portion of the mol. Thus (XIII) is  $\delta$ -nitro-1,2-benzo-[3-sulfonic acid]-5,6-benzo-1'-chloro]-anthraquinone. (4) Compds. containing 2 or more anthracene nuclei are best represented by their structural formulas, rather than by names. [The authors do not always use their own system even in some of their examples; their formulas are also inconsistent. Their suggestion that the substituting nuclei should be indicated by contractions such as Bz, Py, Nt, etc., for  $C_0H_0$ , pyridine and naphthalene, etc., so that (XIII) may also be termed  $\delta$ -nitro-[5,6]-Bz-1-chloro-1,2,5,6-dibenzanthraquinone-[1,2]-Bz-3-sulfonic acid, would create great confusion in English. Abstr.]. See following abstr. J. Bishop Tingle.

Identity of Graebe's Isochrysofluorene with Dihydrobenzanthrene. ROLAND SCHOLL AND CHRISTIAN SEER. Chem. Inst., Univ. Graz. Ber., 44, 1671-4.—Graebe's isochrysofluorene" (Ber., 27, 953 (1894)) formula (I) below, is not C<sub>17</sub>H<sub>12</sub>, but C<sub>17</sub>H<sub>14</sub> and is identical with dihydrobenzanthrene (II) (see preceding abstr.); m. 81°, not

$$(I) \qquad \qquad \begin{pmatrix} H_{i} \\ H_{i} \end{pmatrix}$$

76° (Graebe). The hydrocarbon specimens were identified by direct comparison and also by means of their picrates. When  $\alpha$ -benzylnaphthalene vapor is passed over glowing pumice, in a current of CO<sub>2</sub>, the products consist chiefly of naphthalene,  $\alpha$ -benzylnaphthalene and of "isochrysofluorene," dihydrobenzanthrene (II). The formation of violanthrone may be used as a test for the identification of benzanthrone.

J. BISHOP TINGLE.

Holo- and Merriquinoidal Salts of Benzidine. Reply to the Criticism of Piccard. Walter Madelung. Charlottenburg. Ber., 44, 1674-6.—Piccard's criticism (C. A., 5, 2822) that the author had overlooked the substituting action of Br on the nucleus is unfounded. He had considered it and his published results show that it does not take place.

J. BISHOP TINGLE.

Action of Carbon Disulfide and Alkali Hydroxide on p-Tolyl and on α-Thienyl Methyl Ketone. C. KELBER AND A. SCHWARZ. Pharm. Chem. Inst., Univ. Erlangen. Ber., 44, 1693-700.--p-Tolyl methyl ketone, CS, finely divided KOH and H<sub>2</sub>O (2 drops) when b., give a compound, MeC,H,COCH: C(SH),; lustrous, yellow plates from petroleum ether, m. 84-5°. Yield, 16%. Dimethyl ether, C, H,O(SMe), from MeONa, MeOH and MeI; light yellow, lustrous, highly refractive needles, m. 104-5°. Yield, quant. Dibenzyl ether, C10H2O(SCH2Ph)2, from benzyl bromide; broad, yellow needles from ligroin, m. 111.5-2.5°; mol. wt., in C4H4, 387.2-405.1. Yield, quant. Dibenzoyl derivative, C10H2O(SBz)2, by the Schotten-Baumann method; light yellow crystals from AcOEt + petroleum ether, m. 125°; yield, quant. At 125°, alc. KOH hydrolyzes MeC<sub>4</sub>H<sub>4</sub>COCH: C(SH)<sub>2</sub>; the products, after acidification, consist of H<sub>2</sub>S, AcOH and p-toluic acid. Alc. NH<sub>21</sub> at 140°, in place of KOH, gives NH<sub>4</sub>SCN and p-tolyl methyl ketone. This ketone, together with ethyl-mercaptan, is also produced by the action of alc. HCl on MeC<sub>2</sub>H<sub>4</sub>COCH: C(SH)<sub>2</sub>. At 60°, during 1 hr., PhNH<sub>2</sub> and MeC<sub>2</sub>H<sub>4</sub>COCH: C(SH)<sub>2</sub> form a compound, MeC<sub>2</sub>H<sub>4</sub>COCH: C(SH)NHPh; aggregates of light yellow needles from AcOEt + petroleum ether, m. 80.5-1.5°. A second compound, is formed together with the preceding substance, its proportion being increased if the heating be prolonged; bundles of red needles, m. 192.5-3°. It contains N and S. Under the conditions described above,  $\alpha$ -thienvl methyl ketone, CS, and KOH form the compound, C<sub>2</sub>H<sub>6</sub>OS<sub>2</sub>; yellow plates from petroleum ether, m. 90-1°; mol. wt., in C<sub>8</sub>H<sub>4</sub>, 204.3-6.4. Yield, about 65% of the ketone. Dimethyl ether, C<sub>7</sub>H<sub>4</sub>OS(SMe)<sub>2</sub>, from the mercaptan, MeI and Na (?MeOH); opaque, yellow needles from petroleum ether, m. 96.5°. Yield, quant. Dibenzoyl derivative, C7H4OS(SBz), by the Schotten-Baumann method; thin, yellow, interlaced needles form AcOEt + petroleum ether, m. 118.5°. It becomes highly electrified when rubbed; yield, quant. Alc. KOH, at 125°, converts C<sub>7</sub>H<sub>6</sub>OS<sub>2</sub>, into thiophene-α-carboxylic acid; with alc. NH<sub>2</sub>, at 140°, or alc. HCl, at 130°, \(\alpha\)-thienyl methyl ketone is produced. The above results show that p-tolyl and  $\alpha$ -thienyl methyl ketones react like acetophenone with CS, and KOH. The compds. described below were obtained from the acetophenone deriv., BzCH: (SH)<sub>2</sub>. 2-Phenacylidenetrimethylene 1,3-disulfide formula (I) below, is prepared from ethylene bromide and EtONa; long, pale yellow needles from ligroin, m. 80°; yield, quant. In conc. H2SO4 the color is blue to cherry-red; dil. HNO2 produces a deep red zone. In a current of steam (I) is reduced by Zn dust and aq. NaOH to phenyl ethyl ketone and ethylenemercaptan. 2-Phenacylideneletramethylene 1,3-di-

$$BzCH : C \left\langle \begin{array}{c} S \cdot CH_3 \\ | \\ S \cdot CH_3 \\ \end{array} \right\rangle BzCH : C \left\langle \begin{array}{c} S \cdot CH_3 \\ \\ S \cdot CH_3 \\ \end{array} \right\rangle CH_3$$

sulfide (II), is prepared in a similar manner to (I), from trimethylene bromide; opaque, yellow needles from ligroin, m. 52-3°. Its color reactions are the same as those of

(I). When reduced in the manner described it forms trimethylenemercaptan and phenyl ethyl ketone.

J. BISHOP TINGLE.

Explanation of the Reaction between Quinone and Hydrochloric Acid. Julius Schmidlin. Chem. Lab., Swiss Polytech., Zurich. Ber., 44, 1700-5.—In H<sub>2</sub>O, or Et<sub>2</sub>O, hydroquinol and chloroquinone ppt. in pure quinhydrone containing 1.1—85% of Cl. A similar result is obtained with chlorohydroquinol + quinone, in CHCl<sub>2</sub>, or Et<sub>2</sub>O. In view of these facts and of those discovered by others, it is suggested that the interaction of quinone and HCl includes the following steps. Quinone and HCl give chlorohydroquinol which forms an equilibrium with quinone, chloroquinone and hydroquinol. The sparing sol. of quinhydrone (from quinone and hydroquinol) displaces the equilibrium. The quinhydrone and HCl form hydroquinol and chlorohydroquinol. In dry C<sub>2</sub>H<sub>4</sub>, quinone and chlorohydroquinol ppt. chloroquinhydrone, followed by quinhydrone. When the chloroquinhydrone is dissolved by warming, the liquid, when cold, deposits quinhydrone. Chloroquinone is light lemon-yellow, not yellowish red. The "streak" color of quinhydrone is bluish violet, that of dichloroquinhydrone reddish violet.

Chemical Nature of Allochlorophyll. L. MARCHLEWSKI AND J. MARSZALEK. Cracow. Ber., 44, 1705-8.—The leaves of "ahorns," a plant of Russian Poland, are extremely rich in allochlorophyllan; 26.5 kg. of leaves gave 20 g. of crude chlorophyllan, from which 5.764 g. of allochlorophyllan were sepd. It contained 3% of MeO, showing that from the material obtained previously, at least 1% of the MeO had been removed by the prolonged action of the Zn(OH)<sub>2</sub>. Chlorophyllan contains about 31.8% of phytol; this is but little less than Willstätter (C. A., 4, 1471-2; 5, 872) found in preps. from crude chlorophyll. A constancy in the % of phytol is no evidence as to the homogeneousness of the chlorophyllan. At 105°, allochlorophyllan undergoes a change, and gives hydrolysis products which are insol. in Et<sub>2</sub>O. The allochlorophyllanic acids, produced by hydrolysis of allochlorophyllan which has been dried under reduced pressure, dissolve in Et<sub>2</sub>O. These acids are cherry-red; chlorophyllanic acids are green, or olive-green. Zinc allochlorophyll is prepared from allochlorophyllan and Zn(OAc), in alc.; it resembles zinc chlorophyll, but exhibits a different spectrum. With alkalies it gives a substance similar to alkachlorophyll. With conc. HCl, Zn allophyll fails to form any substance similar to phyllotaonin and it is doubtful if its further degradation would produce phylloporphyrins. Like chlorophyll, allochlorophyll contains CO, Me and a CO, H in combination with the phytol complex, but the colored nuclei of the 2 compds. are very different. It is quite probable that Willstätter's chlorophyllase may eliminate phytol from allochlorophyll, and may esterify the colored portion of the mol. This would agree with Tzvyet's statement, that cryst. chlorophyll is a mixt. J. BISHOP TINGLE.

o- and m-Iodo-p-Cresyl Methyl Ether and Derivatives with Polyvalent Iodine. C. WILLGERODT AND RUDOLPH SCHLOSS. Freiburg i/B. Ber., 44, 1708-11.—In glacial AcOH, ICl and p-cresyl methyl ether give a mixt., which, after prolonged treatment with SO<sub>2</sub>, is sepd. by means of alc. The more sol. portion consists of 3-iodo-4-methoxytoluene (b. 237-8°). The other substance is 2-iodo-4-methoxytoluene, MeOC<sub>6</sub>H<sub>2</sub>IMe; white, rhombic plates from alc., m. 75°. 4-Methoxytoluene-3-iododi-chloride, MeOC<sub>6</sub>H<sub>2</sub>MeICl<sub>2</sub>, from the 3-iodo compd. and Cl, at o°; yellow, very unstable needles. With Ph<sub>2</sub>Hg and H<sub>2</sub>O, followed by KI, it gives 4-methoxytoluene-3-phenyl-iodinium iodide, MeOC<sub>6</sub>H<sub>2</sub>MeIIPh; pale yellow needles from alc., m. 181°. 4-Methoxy-toluene-2-iododichloride, is prepared like its isomer; orange-yellow, interlaced needles, decomp. 58-60°. It is very stable. 4-Methoxy-2-iodosotoluene, MeOC<sub>6</sub>H<sub>2</sub>MeIO, from the preceding compd. and aq. NaOH; pale yellow, amorphous powder, explodes 176° (decomp.). Acetate, MeOC<sub>6</sub>H<sub>2</sub>MeIOAc, from the iodoso deriv. and Ac<sub>2</sub>O; long,

silky lustrous crystals, m. 120-2°. 4-Methoxytoluene-2-phenyliodinium iodide is prepared like the isomer above; almost colorless needles from alc., m. 166°.

J. BISHOP TINGLE.

Synthesis of Imineazole Derivatives. A. WINDAUS AND H. OPITZ. Med. Dept. Lab., Univ. Freiburg i/B. Ber., 44, 1721-5.—Imineazylacetkydraside, formula (I) below, is prepared by b. imineazylacetic ester with abs. alc. and N<sub>2</sub>H<sub>4</sub>·H<sub>2</sub>O; bundles

of needles from abs. alc., m. 189° (decomp.). Yield, over 80%. Dihydrochloride, prisms, m. 230°. When treated, at the ordinary temp., with abs. alc. and amyl nitrite and the product b. and subsequently hydrolyzed by means of abs. alc., imineazylmethylamine dihydrochloride (II) is formed; long, thin prisms from MeOH, softens 236°. Chloroplatinate, C.H., N., H.PtCl., opaque, rhombic plates, blackens 288°. Dipicrate, deep yellow, highly lustrous, hexagonal plates from H.O. m. 209°. Picrolonate, C<sub>2</sub>H<sub>2</sub>O<sub>1</sub>, N<sub>11</sub>, bundles of long, yellow needles from H<sub>2</sub>O, m. 273° (decomp.). β-Imincazylethyl. alcohol phosphotungstate is prepared from \(\beta\)-imineazylethylamine hydrochloride, Ba(NO<sub>2</sub>), and H<sub>2</sub>O, the product being treated first with H<sub>2</sub>SO<sub>4</sub>, then with phosphotungstic acid; short, thick prisms from H<sub>2</sub>O. Yield, 211% of the hydrochloride. It was decomp. by means of Ba(OH), and the clear soln. used for the production of the derivatives described below. Chloroplatinate, (C.H.ON.), H.PtCla; well-developed, orange-yellow needles from alc. + Et<sub>2</sub>O, m. 175°. Yield, about 16% of the phosphotungstate. Hydrochloride, aggregates of needles from H<sub>2</sub>O. Picrolonate, light yellow needles, m. 264° (decomp.). Nitroimineazolecarboxylic acid possibly (III), is formed by b. crude  $\beta$ -imineazylethyl alc. with HNO, (25%); long,

almost colorless prisms from  $H_1O$ , m. above 300° (decomp.). The compd. has a strongly acid reaction, is sparingly sol. in dil. acids, but dissolves readily in aq. alkalies, the solns. being deep yellow.

J. BISHOP TINGLE.

Degradation of Indanthrene to Dihydroxypyrazinoanthraquinone and its Behavior towards Benzoyl Chloride and Sodium Alcoholate. ROLAND SCHOLL AND SIEGFRIED EDLBACHER. Chem. Inst., Univ. Graz. Ber., 44, 1727-37.—Indanthene, formula (I) below (R-H), purified by means of b. conc. HCl and glacial AcOH, was b. during 40 hrs.

with glacial AcOH and CrO<sub>3</sub> (4 pts.), which was divided into 5 portions, each being added at intervals of 8 hrs. The clear filtrate deposits  $\alpha, \beta$ -dihydroxy-1,2-pyrazino-

anthraquinone (α,β-dihydroxyanthraquinoxalinequinone) (II); crystals from PhNO<sub>4</sub>, sublimes about 300°, chars about 370°. In PhOH, or quinoline the color is brownish yellow, in conc. H<sub>2</sub>SO<sub>4</sub>, red. It is not changed by b. conc. H<sub>2</sub>SO<sub>4</sub>, nor by Br, at 100°, during 10 hrs. Yield, 50% of (I). Disodium salt, C1eH2O4N2Na, from EtONa, abs. alc. and (II); reddish brown. With alkaline Na hyposulfite (II) gives a brownish red vat dye, at the ord. temp.; it has but little affinity for vegetable fibers. Distillation of (II) with Zn dust converts it into a compd., probably 1,2-pyrasinoanthracene; brown, its soln. in C<sub>a</sub>H<sub>a</sub> has a blue fluorescence. Nitro-a,β-dihydroxy-1,2-pyrazinoanthraquinone, C<sub>10</sub>H<sub>2</sub>O<sub>2</sub>N<sub>3</sub>, from (II) and HNO<sub>2</sub> (d. 1.52), at the ordinary temp.; pale yellow. Sodium salt, red. Colorless (NH<sub>4</sub>)<sub>2</sub>S reduces the nitro compd. to amino-α,β-dihydroxy-1,2pyrazinoanthraquinone, C1eHeO4N2; violet, microscopic needles from PhNO2, m. about 365°. In pyridine, quinoline, or PhNO, the color is red to violet, in hot xylene green, in conc. H.SO, yellow, and in aq. NH, violet. At 170°, anhydrous C.H.O, and 1,2aminoanthraquinone (III) form (II), thereby verifying the formula given. N-Dibenzoylindanthrene (I) (R = Bz), is prepared by b. cryst. or amorphous indanthrene with BzCl (70 pts.), during 1 hr., treating the product with Na<sub>2</sub>CO<sub>2</sub> and steam distilling to remove BzOH; red, microscopic needles from xylene; yield, almost quant. It is also obtained by b. indanthrene with Bz<sub>2</sub>O. In ordinary solvents the color is yellow to brownish red; in BzCl blue, changing to greenish brown when heated; in conc. H,SO4, brownish yellow. The benzoyl deriv. is hydrolyzed to indanthrene immediately by alc. KOH, or by conc. H.SO., thus establishing it as a N-Bz deriv. In b. BzCl, Cl converts N-dibenzoylindanthrene into chloroanthraquinoneasine dichloride; dark green needles from PhNO2. When b. with quinoline it gives chloroindanthrene; blue. At the ordinary temp., abs. MeOH and MeONa convert (I) (R = H) into an additive product (IV), in which the positions of the ONa and OMe groups are arbitrary; the

compd. is purified by washing with abs. MeOH and abs. MeOH + abs.  $Et_2O$ ; blackish blue powder. With  $H_2O$  indanthene is immediately regenerated. EtONa + abs. alc. give a corresponding addition compound, Under similar conditions to those just described, anthraquinoneazine, MeOH and MeONa form an additive compound,  $C_{22}H_{13}O_4N_3$ -2MeONa; green, unstable powder. It is decomp. slowly by MeOH into anthraquinoneazhydrine. Alc. and EtONa react in a similar manner, but the Na compd., when decomp. by alc., gives some indanthrene. It is evident, from these results, that a portion only of the anthraquinoneazine forms the additive compd., the remainder being reduced. Anthraquinone is not changed by Na alcoholates, under the conditions described.

I. BISHOP TINGLE.

Ring Production in the Peri Position in the Maphthalene Series. III. Derivatives of o,p-Dinitrophenyl-1-amino-8-naphthylamine. Frans Sachs and R. B. Forster. Chem. Inst., Univ. Berlin. Ber., 44, 1738-48; see C. A., 3, 1870, 1982.—2,4-Dinitrophenyl-1-perimidine formula (I) below, is prepared by b. 2,4-dinitrophenyl-1-amino-

8-naphthylamine with anhydrous HCO,H; reddish brown crystals from dil. MeOH,

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m. 175°, decomp. 265°. In conc. H.SO<sub>4</sub> its color is brown. It is insol. in alkalies. Yield, 95% of the amine. The name perimidine is applied to the complex (II), from 1,8-naphthylenediamine and HCO<sub>2</sub>H. Picrate, C<sub>22</sub>H<sub>12</sub>O<sub>11</sub>N<sub>2</sub>, orange-red crystals from alc., m. 232°. 2,4-Dinitrophenyl-1-amino-8-acetylaminonaphthalene, AcNHC10Ha-NHC<sub>2</sub>H<sub>2</sub>(NO<sub>2</sub>)<sub>2</sub>, is prepared in a similar manner to (I), from Ac<sub>2</sub>O in place of HCO<sub>2</sub>H; yellow crystals from alc., m. 237°. In conc. H.SO, the color is dark yellow, when warmed the liquid becomes colorless. Yield, 81% of the amine. At the ordinary temp., conc. HNO, converts it into a tetranitro compound; yellow crystals, m. 248°. 2,4-Dinitrophenyl-I-amino-8-benzoylaminonaphthalene, BzNHC10HaNHC4Ha(NO2)2 from BzCl and the amine; crystals from glacial AcOH, or xylene, m. 271-2°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is amber-yellow, it becomes much lighter when the liquid is warmed. 2,4-Dinitrophenyl-1-amino-8-cinnamoylaminonaphthalene, PhCH: CHCONH-C<sub>10</sub>H<sub>e</sub>NHC<sub>2</sub>H<sub>e</sub>(NO<sub>2</sub>)<sub>2</sub>, from cinnamyl chloride, at 100°; yellow crystals from xylene, m. 258-9°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is amber-yellow, when warmed it becomes color-2,4-Dinitrophenyl-1-amino-8-carbethoxyaminonaphthalene, HC<sub>2</sub>H<sub>2</sub>(NO<sub>2</sub>), by heating the amine with Et chlorocarbonate, at 100°, during 17 hrs.; orange-colored crystals from CHCl<sub>2</sub> + petroleum ether, m. 184-5°, decomp. 260-70°. Yield, 110% of the amine. At 193° (10-12 min.), it is converted into alc. and 2,4dinitrophenyl-1-dihydro-2-perimidone (III); red, triangular prisms with 0.5 CaH10,

from xylene, darkens about 150°, m. 267-8° (decomp.). Yield, about 64% of the ester. In conc. H<sub>2</sub>SO<sub>4</sub> the color is light yellow, changing to red when warmed. In presence of glacial AcOH and AcONa, COCl<sub>2</sub> and the amine react slowly to form (III). 2,4-Dinitrophenyl-1-amino-8-phenylthiocarbamylaminophthalene, PhNHCSNHC<sub>10</sub>H<sub>0</sub>NH-C<sub>0</sub>H<sub>1</sub>(NO<sub>2</sub>)<sub>2</sub>, from Ph isothiocyanate, in CHCl<sub>2</sub>; lustrous, orange-red crystals from CHCl<sub>2</sub>+petroleum ether, m. 182°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is dark yellow, changing to red when heated. Yield, 100% of the amine. Methyl 2,4-dinitrophenyl-1-amino-8-naphthyloxamidate, MeO<sub>2</sub>CCONHC<sub>10</sub>H<sub>2</sub>NHC<sub>0</sub>H<sub>2</sub>(NO<sub>2</sub>)<sub>2</sub>, from the amine and di-Me oxalate, at 100°, during 7 dys.; yellow, flocculent crystals from acetone + H<sub>2</sub>O<sub>2</sub>, m. 209-10. Yield, 115% of the amine. Ethyl ester, C<sub>20</sub>H<sub>16</sub>O<sub>7</sub>N<sub>4</sub>, is prepared in a similar manner to the preceding compd.; yellow crystals from AcOEt, m. 191-2°. Ethyl 2,4-dinitrophenyl-2-perimidinecarboxylate (IV), is formed together with the preceding compd.

$$C_{10}H_{\bullet} \left\langle \begin{array}{c} N \\ N[C_{\bullet}H_{\bullet}(NO_{\bullet})_{\bullet}] \end{array} \right\rangle CCO_{\bullet}Et \qquad C_{10}H_{\bullet} \left\langle \begin{array}{c} N \\ N[C_{\bullet}H_{\bullet}(NO_{\bullet})_{\bullet}] \end{array} \right\rangle CCH_{\bullet}CH_{\bullet}CO_{\bullet}H$$

and is sepd. by its greater solubility; red crystals, m. about 171-2°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is brown. 2,4-Dinitrophenyl-1-amino-8-naphthylsuccinamidic acid, (O<sub>2</sub>N)<sub>2</sub>C<sub>2</sub>H<sub>2</sub>NHC<sub>10</sub>H<sub>2</sub>NHCOCH<sub>2</sub>CO<sub>2</sub>H, from the amine and succinic anhydride, in glacial AcOH, at the b. p., during 15 min.; pale yellow, cryst. powder with 1 C<sub>2</sub>H<sub>4</sub>O<sub>3</sub> from dil. alc., becomes red 190°, m. 227° (gas evol.). In alkalies, or conc. H<sub>2</sub>SO<sub>4</sub> the color is red. If the b. of the mixture as above be prolonged during at least 4 hrs., the

product deposited from alc. consists of a red, cryst. powder, m. 227°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is greenish yellow. The compd. is probably the *perimidine derivative* (V). Yield, about 110% of the amine. The substance dissolves in warm, but not in cold aq. alkalies; from C<sub>2</sub>H<sub>4</sub> + CHCl<sub>6</sub> it deposits in large, yellow crystals, containing 0.5 C<sub>2</sub>H<sub>4</sub>. In b., glacial AcOH the amine and phthalic anhydride form 1-[2,4-dinitro-phenyl]-2-[2-carboxyphenyl]perimidine (VI); yellow crystals from glacial AcOH, or

$$C_{10}H_{4} \underbrace{\begin{array}{c} N \\ N[C_{6}H_{4}(NO_{2})_{2}] \end{array}}_{CC_{4}H_{4}CO_{2}H}$$

$$C_{10}H_{4} \underbrace{\begin{array}{c} N \\ N[C_{4}H_{4}(NO_{2})_{2}] \end{array}}_{CH} CH_{4}$$

$$(VI)$$

pyridine, m. 297°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is dark blue, changing to red when heated. Glacial AcOH and Zn dust reduce (VI) to a diamine, not m. 240°. Picrate, m. 220°. In conc. H.SO, the color is brown, becoming redder when heated. At 60°, in presence of glacial AcOH and dil. HCl, HCHO and 2,4-dinitrophenylnaphthylenediamine give a compound, possibly (VII); it is purified by treatment with PhNH, and pyridine; red powder; insol. in alkalies, or dil. acids. In conc. H.SO. the color is dark amber, when warmed the soln. chars. Yield, 80% of the amine. 2,4-Dinitrophenyl-1-amino-8naphthyliminoacetone, (O2N)2C2H2NHC10H2N: CMe2, from the amine and acetone: yellow crystals from CHCl<sub>2</sub> + petroleum ether, m. 166-7°. It is resolved into its components by warming with dil. alc. 2,4-Dinitrophenyl-1-amino-8-naphthylaminoacetophenone, (O<sub>2</sub>N)<sub>2</sub>C<sub>2</sub>H<sub>2</sub>NHC<sub>10</sub>H<sub>2</sub>N: CPhMe, from acetophenone, at 100°, during 18 hrs.; crystals (red changing to yellow) from amyl alc., m. 163-4°. Ethyl 2,4-dinitrophenyl-1-amino-8-naphthyliminoacetoacetate, (O2N)2C2H2NHC10H2N: CMeCH2CO2Et, prepared in a similar manner to the preceding compd., from Et acetoacetate; it is also formed slowly from the components at the ordinary temp. Large crystals from CHCl. + petroleum ether, m. 167-8°. It is hydrolyzed by AcOH. J. BISHOP TINGLE.

New Method for the Preparation of Acenaphthenequinone from Acenaphthene. Arnold Reissert. Lab., Univ. Marburg. Ber. 44, 1749-52.—When treated with b. amyl nitrite (4 mol.) and dry HCl, acenaphthene is converted into a mixt. of acenaphthenequinone oximes, which are removed by soln. in dil., aq. NaOH. Yield, 75.5%. The same products, but in smaller yield, are formed by passing the vapor of amyl nitrite and dry HCl into a b. alc. soln. of acenaphthene. The oximes are sepd. by prolonged b. with dil., aq. Na<sub>2</sub>CO<sub>2</sub>, the portion which dissolves the more readily consists of the ordinary oxime, as prepared from HONH<sub>2</sub> and acenaphthenequinone; m. in part and darkens 220°, becomes solid then m. 290-300°. In the literature it is stated to m. 230°. When heated on a H<sub>2</sub>O-bath, during 1 hr., with H<sub>2</sub>SO<sub>4</sub> (7 pts.; 75%), it is converted into acenaphthenequinone, m. 262-3°. The isomeric oxir e is deposited from glacial AcOH in small, soft, pale yellow crystals, decomp. and explodes feebly 207°. Prolonged b. with glacial AcOH transforms it into the isomer, m. 220°. Hot H<sub>2</sub>SO<sub>4</sub> converts it chiefly into naphthalimide, or its decomp. products, NH<sub>2</sub> and naphthalic acid. The nature of the isomerism of the oximes is unknown. J. B. T.

Action of Halogenated Aliphatic Acidic Halides on Malonic Ester. II. Synthesis of Tetramic Acid. Erich Benary. Chem. Inst., Univ. Berlin. Ber., 44, 1759-65; see C. A., 1, 1544.—Me tetrone- $\alpha$ -carboxylate formula (I) below, is prepared from Me sodiomalonate and chloroacetyl chloride, in Et<sub>2</sub>O; m. 188-9°, not 171-3°; yield, about 65%. Copper salt, Cu(C<sub>8</sub>H<sub>8</sub>O<sub>8</sub>)<sub>2</sub>, yellowish green; decomp. about 220°. Ethyl ester

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copper salt,  $Cu(C_7H_7O_8)_{27}$  light blue. Ethyl  $\gamma$ -methyltetrone- $\alpha$ -carboxylate (II), is prepared from bromopropionyl bromide and Et sodiomalonate (2 mols.) and is purified by means of its Cu salt; colorless plates from  $H_2O_7$ , softens  $85^\circ$ , m.  $89-90^\circ$ . Copper salt,  $Cu(C_9H_9O_8)_{27}$  bluish green powder. The ester is hydrolyzed by aq.  $Ba(OH)_{27}$  the resulting barium salt, when treated with dil. HCl, decomp. into  $CO_2$  and  $\gamma$ -methylestronic anhydrids (III); hard aggregates of quadratic plates from  $H_2O_7$ , softens  $112^\circ$ , m.  $117-9^\circ$ . In  $H_2O$  it gives a red color with FeCl<sub>2</sub> and a violet with  $NaNO_2$ .  $\alpha$ ,  $\alpha$ -Dicarbethoxy- $\gamma$ -allene oxide (V), which is formed together with tetrone- $\alpha$ -carboxylic ester, is converted by dry  $NH_2$  and  $Et_2O_7$  into the amide. Yield, about 30%. b.

alc. KOH hydrolyzes it to tetramyl-a-carboxylic acid (IV); small needles from H<sub>1</sub>O, darkens about 200°, decomp. 214°. Dipotassium salt, C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>NK<sub>2</sub>, from alc. KOH, sparingly sol. The acid reduces warm, aq. Ag-NH<sub>2</sub> solns. When b. with H<sub>2</sub>O (IV) evolves CO<sub>2</sub> and gives tetramol (VI); flat, lustrous prisms from MeOH, softens and darkens about 200°, m. 211°; mol. wt., in freezing glacial AcOH, 105. It reduces warm, aq. Ag-NH<sub>2</sub> soln., has a neutral reaction, does not give a ppt. with dry NH<sub>3</sub> in C<sub>2</sub>H<sub>4</sub> and evolves NH<sub>2</sub> when b. with aq. NaOH, or Ba(OH)<sub>3</sub>. Aq. FeCl<sub>2</sub> produces a blood-red color. Isonitroso derivative (VII), from (VI), NaNO<sub>2</sub> and aq. HCl, at 0°; small, light blue, microscopic plates, becomes lighter in color about 160°, darkens and decomp. explosively 205°. In H<sub>2</sub>O the color is violet, in aq. NH<sub>3</sub>, or Na<sub>2</sub>CO<sub>3</sub>, wine-red. It is decomp. by dil. aq. NaOH.

Perchlorates of the Choline and Neurine Series: Detection of Choline and Neurine. K. A. HOFMANN AND K. HÖBOLD. Inorg. Lab., Techn. Hochsch., Berlin. Ber., 44, 1766-71.—Choline perchlorate, C2H2NCH2CH2OHClO4, is deposited in aggregates of leaslets, by adding HClO<sub>4</sub> (70% soln.) to the base in abs. alc.; polyhedral crystals are obtained from H<sub>2</sub>O, softens 270°, m. 273°. At 15°, 100 pts. of H<sub>2</sub>O dissolve about 290 pts. of the salt; mol. wt., in H<sub>2</sub>O, 108-10. The same salt is obtained from choline prepared by the hydrolysis of egg yolk, but the perchlorate is too readily sol. to be used as a test for choline. When evapd. on the  $H_2O$ -bath with  $HNO_2$  (65%), or treated at 15° with pure HNO<sub>2</sub>, the perchlorate is converted into the nitric ester, C<sub>2</sub>H<sub>2</sub>NCH<sub>2</sub>-CH<sub>2</sub>NO<sub>2</sub>ClO<sub>4</sub>; long, thin, rectangular, lustrous plates of the mono-, or triclinic system, m. 185-6°, explodes when strongly heated. At 15° and 20°, 100 pts. of H<sub>2</sub>O dissolve o.62 and o.82 pts., resp. The compd. gives the ordinary color reactions for nitric esters and is well adapted for the detection of choline. The ester salt is not decomp. by recryst. from aq. HClO4, or NH2. Neurine perchlorate, C2H2NCH: CH2ClO4, is prepared in a similar manner to the choline salt; thick, rhombic prisms; it reduces "permanganate," in presence of NaHCO2, at the ordinary temp. At 14.5°, 100 pts. of H.O dissolve 4.89 pts. of the salt. Dibromoethyltrimethylammonium perchlorate, C<sub>2</sub>H<sub>2</sub>NC<sub>2</sub>H<sub>2</sub>Br<sub>2</sub>ClO<sub>4</sub>, is prepared by brominating neurine and adding HClO<sub>4</sub> to the product; large, colorless, doubly refractive plates. At 15°, 100 pts. of H<sub>2</sub>O dissolve 2.2 pts. of the salt. Bromovinyltrimethylammonium perchlorate, C<sub>2</sub>H<sub>2</sub>NCBr: CH<sub>2</sub>ClO<sub>4</sub>, is formed by b. dibromoneurine with AgNO<sub>8</sub> soln. and adding HClO<sub>4</sub> to the product; long, pointed, lustrous plates. It reduces "permanganate" + NaHCO, soln. immediately. At 14°, 100 pts. of H<sub>2</sub>O dissolve 2.65 pts. of the salt. J. B. T.

Keto-enolic Equilibrium of Acetoacetic Ester. A. HANTZSCH. Ber., 44, 1771-6.

—In connection with the papers of K. H. Meyer (C. A., 5, 2624) and of L. Knorr (C. A., 5, 2836), the author calls attention to the fact that, in his previous paper (C. A., 5, 504, 705), the results given for the equil. content, under varying conditions, are incorrect.

In his calculations the reciprocal values of the absorption became inadvertently substituted for the figures expressing the absorption. The method used in the cal. of the equil, point is given in full. The following numbers represent the % of enolic Et acetoacetate in the resp. solvents; the numbers in italics are Meyer's values and those in parentheses reproduce Knorr's results: In hexane, 54, 46.4, (31). In Et.O, 44, 27.1, (9.5). In alc., 13, 12, (-). In MeOH, 6, 6.9, (-). In H<sub>2</sub>O, 0.1, 0.4, (-). In dil. HCl, o.o, o.3, (-). The differences in the values found in hexane and Et.O are probably due, at least in part, to the fact that the author used much more dil. solns. than the others. Other errors are undoubtedly due to catalytic effects, especially those caused by traces of H.O from the air. These factors would doubtless influence the Et.O soins. to the greatest extent. A recalc. of the temp. coeff. of Et acetoacetate, in H<sub>2</sub>O, indicates that at 20° the % of enolic ester is about 0.14, at 80° 0.5. Meyer's value, at 20°, is 0.4%. Knorr is in error in attributing to the author the statement that the homogeneous ester "is almost completely enolic." What the author did say was that, contrary to the conclusions of Brühl, the homogenous ester" could not possibly consist only of the keto form." I. BISHOP TINGLE.

Molecular Condition of Organic Ammonium Halides in Nondissociating Media. A. HANTZSCH AND O. K. HOFMANN. Ber., 44, 1776-83.—Abs. CHCla is prepared, without difficulty, by shaking the com. product with conc. H<sub>2</sub>SO<sub>4</sub> during about 15 mins.; it is then washed repeatedly with dil. aq., NaOH and finally with H<sub>2</sub>O, and dried by contact with recently ignited K<sub>2</sub>CO<sub>2</sub> in bottles which are almost completely filled. The product may be used directly without previous distillation. The constant, for the b. p. method, is taken as 39.0. Me, NCl, Me, NBr, Me, NI, EtNH, Cl and PrNH, Cl, dissolve too sparingly in CHCl, to permit of detg. their mol. wts. The numbers which follow the formulas given below, express the resp. mol. wts. found in b. CHCl, the numbers in parentheses represent approx. what multiple of the formula wt. - this mol. wt. Pr.NI 1560-613 (5); Pr.NCl, 573-6 (2-3); Et.NHCl, 202-97 (2); Et.NH.Cl, 257-534 (3-5); Me<sub>2</sub>NH<sub>2</sub>Cl, 258-429 (3-5); isoamylammonium chloride, too great for determination. The variable results in the case of Et,NHCl, Et,NH,Cl and Me,NH,Cl are due, in part, to the presence of traces of H<sub>2</sub>O, because the salts in question are very hydroscopic. This also accounts for the values obtained previously for Me, NH, Cl, according to which it was tri- and tetramol. Isoamylammonium chloride crystallizes readily from CHCl, and the solns. exhibit no sign of being colloidal. It is, therefore, remarkable that the effect of the salt on the b. p. of the soln. should be so vanishingly small. These results show that the association of the alkylammonium salts is not dependent, to any marked degree, on the mol. wt.; it diminishes as the number of alkyls increases and it is smaller in the case of the chlorides than in that of the iodides. This same effect is also shown in the results given below for the N-methlyphenylacridonium salts mentioned, the numbers have the same significance as before. Chloride, 599-817 (2-3); bromide, 790 (2); thiocyanate, 1075-405 (3-4); iodide, 1952-2240 (5-6). The colors of the above solns. are greenish yellow, light red, red and deep red, resp. The following results were obtained with the phenylacridonium salts mentioned. Chloride, 374-84 (1-2); iodide, 502-55 (1-2). The colors of the solns, are yellowish brown and light red, resp. The following additional values were detd. in the case of the N-methylphenylacridonium salts mentioned. In b. pyridine (K 29.5); iodide, 381-408 (1). The soln. is red. In freezing PhOH (K 72.0); iodide, 371-436 (1); thiocyanate, 297-355 (1). The iodide soln. is yellow when dil., brownish when conc.; that of the thiocyanate is yellow. I. BISHOP TINGLE.

Chromoisomerism of Pyridine, Quinoline and Acridine Salts and its Significance as Valence Isomerism. A. HANTZSCH. Ber., 44, 1783-828.—The following phenylacridonium salts have the colors mentioned: perchloride, yellow, green; nitrate, yel-

lowish red; acid sulfate, yellow, red, green; chloride, brownish yellow, CHCla compd. yellowish brown; bromide, brownish yellow; iodide, brownish yellow, CHCl, compd. red. N-Methylphenylacridonium salts are colored as follows: perchlorate, nitrate and acid sulfate, yellow; nitrite and bromide, brownish yellow; sulfite, yellow, red, green, brown; fluoride, yellow, green; chloride, yellow, green, hydrate, greenish yellow, brown; thiocyanate, orange, green; iodide, red. The author's previous statement that polychromism is only exhibited by halogen salts and occasionally by sulfites, is incorrect; it may obviously occur in the case of salts of other acids, such as H<sub>2</sub>SO<sub>4</sub>, which contain O and are not tautomeric. The conclusion is drawn that the polychromism of the acridonium salts is, in principle, independent of the nature of the anion and cation; these simply influence the conditions of stability to the advantage, or disadvantage of 1 or more of the possible forms. N-Methylphenylacridonium sulfite has the following colors, in the solvents mentioned; the solns, were all conc.; abs. alc., red; alc.  $+ H_0O_1$ greenish yellow; amyl alc., greenish brown; glacial AcOH, red; acetylene diand tetrachloride, green; acetonitrile, golden yellow; nitromethane yellow-The following colors are exhibited by the ish brown; PhNO, brown. resp. N-methylphenylacridonium salts; the numbers give the degree of association in CHCl2; in pyridine and in PhOH the salts are not associated: In CHCl2, chloride greenish yellow (2-3); bromide, light red (2-3); thiocyanate, red (3-4); iodide, deep red (5). In pyridine the colors are greenish brown, yellowish red, light red and deep red, resp. In PhOH, brown, brown, brownish orange and reddish brown, resp. Similar data for phenylacridonium salts are as follows: in CHCla, chloride, yellowish brown (1-2); iodide, light red (1-2). In PhOH (not associated), chloride, light red; iodide, red. These results show that the polychromism, exhibited by the salts in soln., is independent of their association. The intensely yellow solns, of all cotarnine salts in H,O, alc., or CHCl, are optically identical, but in the first the salts are dissociated practically completely; in the second they are undissociated and in the last solvent they exist associated. Therefore the color is not dependent on the dissociation of the salts. The ultraviolet absorption spectra of polychromic solns. of polychromic salts, differs considerably in different solvents. In the case of any solvent, differences in the spectra may be produced by change of temp. or conc, or by variation in the nature of the anion. On the other hand, in sharp contrast to these polychromic compds., colored solns, of nearly related substituted ammonium salts are known whose absorptive power is almost equal. These substances are termed "idiochroms," their absorption curves are not materially affected by change in the solvent, anion, conc., or temp. These statements are illustrated by curves representing the ultraviolet absorption spectra of cotarnine salts. A similar investigation of salts of pyridine, quinoline, isoquinoline and of acridine shows that their solns, contain the chromophores of isomeric ammonium ions, which are in equil. Certain exceptional limiting cases will be considered later. The equil. in soln. of chromoisomeric, substituted ammonium halides is displaced so as to increase the proportion of the more deeply colored isomer, by the following factors: increase in the associative (or better, by decrease in the dissociative) action of the solvent; by increase in the at. wt. of the halogen; by rise in temp., or by increase in conc. of the soln. Beer's law is discussed and shown to be invalid, because the absorption, in many cases, changes with the conc., thus proving that the equil. of the colored compds. must be displaced as the conc. is altered. As the polychromism in soln. has been shown (as above) to be due to isomerism, the same must be true of solids which exhibit polychromism. This is demonstrated by the fact that many of the solns. are identical in color with the solid solvates (hydrates, alcoholates, etc.) which they deposit. It follows, therefore, that the solvent may act simply as a catalyst, to change I form into another, or it may combine, in which case the color of the resulting solvate is independent of its physical state of liquid (soln.), or solid. The chromotropism (color change) of the solid salts is regarded as being essentially chem. in its nature and as consisting in a true rearrangement of an unstable, chromoisomer into a stable form. The acridonium salts appear to form 3 series of homogeneous chromoisomers, viz., the yellow, the green and the red salts. The orange, greenish vellow, brownish yellow, greenish brown and dark brown forms are to be regarded as solid solns., or mixts. of 2 or of all 3 of the homogeneous isomers. It is shown, for example, that the dark brown salt is produced by mixing intimately the green and the red salts. The chromoisomeric quinoline, isoquinoline and pyridine salts are very unstable and, apparently, exist only in 2 forms. First those with the feetler absorption which, in the quinoline series, are almost colorless and in the case of pyridine entirely so. The members of the second series exhibit stronger absorption, in the case of the quinoline derivatives they are dark yellow to orange-red, whereas the pyridine salts are yellow. Chromoisomerism has also been observed in the case of a few salts of cotarnine and of neocotarnine. The constitution and formulation of the above chromoisomers is discussed at considerable length. The suggestion of Tinkler (C.A., 3, 2801)and of H. Decker (C. A., 4, 757) that chromoisomerism is due to changes in structure. such as can be represented by ordinary formulas, is rejected. W. Ostwald's idea that chromoisomerism is colloidal in its nature, is regarded as being opposed to many facts. That the chromoisomeric, substituted ammonium salts are N-stereoisomers, or that they are due to differences in the linkage arrangements of the N-nucleus, somewhat as shown in formulas (I), (II) and (III) below, is considered to be very improbable.

The hypothesis which is adopted is to formulate them as valence isomers. This involves the conclusion that the ammonium ions are not symmetrical; of the 4 groups linked to N, 1 is more feebly attached than the other 3. In the ammonium salts the anion is linked to that definit group which is the more feebly attached to the N at. As the 4 groups linked to the N at. are not equiv., ammonium ions and salts may exist in isomeric fo ms such as (IV) and (V), in which the weaker linkage is represented by a

dotted line. The following additional data are given regarding the salts mentioned above. Phenylacridonium salts: Chloride, yellowish brown crystals; greenish blue in transmitted light. Bromide, brown needles with a green dichroism. Iodide, yellowish brown. Its compd. with CHCl<sub>2</sub> is red. Perchlorate, yellowish brown needles which are dichroic to brownish green; its powder is green. Nitrate, large, orange-red needles of varying shade, from alc. Acid sulfate, yellow needles from dil. aq. H<sub>2</sub>SO<sub>4</sub>, usually with 0.5 H<sub>2</sub>O. The anhydrous salt has the same yellow color. Occasionally brownish yellow needles are deposited; this is a mixed form of the yellow, green and red, in which the first predominates largely. Red and green salts, crystallize as sep. individuals from the same soln. of the acridine in alc. H<sub>2</sub>SO<sub>4</sub>. Small variations of temp., or conc. det. the proportion in which the 2 forms are deposited. N-Methylpheny acridonium salts: Chloride. In addition to the ordinary yellow form, a light green salt is deposited from CHCl<sub>2</sub>, under reduced pressure. The aq. soln. deposits the chromo-isomeric yellowish brown and green monohydrates. Brom de, yellowish brown. Thiocyanate, yellow from H<sub>2</sub>O, becomes orange when dried. Slow cool ng of the yellow

form, in H<sub>2</sub>O, causes the deposition of light orange prisms. When the cooling is rapid yellow, microscopic crystals are formed. A green form was obtained once, from alc. soin. Iodide, deep red. Difluoride, C<sub>80</sub>H<sub>16</sub>NH.2HF, crystals from aq. HF. Recryst. from alc. converts it into the monofluoride, CanHiaNH.HF; it evolves HF and becomes greenish yellow when heated. Nitrate and acid sulfate, yellow. Perchlorate, slender, yellow needles. In pyridine its color is reddish brown, in other solvents yellow. Nitrite, yellowish brown crystals (mixed form). Sulfite, (C<sub>20</sub>H<sub>14</sub>N).H<sub>2</sub>SO<sub>2</sub>, has been isolated in a green, yellow and brownish red (mixture of red and green) forms. The yellow salt is the most labil and is obtained by rubbing the powdered green salt with PhNO, on a porous p'ate, or by pptn. with Et.O from soln. in glacial AcOH, or PhNO. The green salt, into which the yellow passes very easily, cannot be obtained in a pure condition from alc., as stated previously. It is necessary to powder the crystals which are deposited from alc. and treat the powder repeatedly with Et.O. The red salt is deposited in small plates from the green CHCl, soln. It has, apparently, not been obtained in a condition of purity. In absence of CHCl, it passes rapidly into the brown salt, which is a mixture of the green and red forms; it is also produced from phenylmethylacridol and SO<sub>2</sub>. N-Ethyl- benzyl- and allylphenylacridonium iodides are formed with some difficulty from the alkyl iodides. The iodides are all red and the Et deriv. is pentamol. in CHCl<sub>2</sub>. Ethylphenylacridonium chloride is yellowish green. The other salts of these compds. resemble those described above, consequently it may be stated that the chromoisomerism of the quaternary phenylacridonium salts is hardly influenced by the nature and mass of the entrant hydrocarbon radicles. This indicates that the affinity effects of the satd. and unsatd. alkyls are very similar, but that they differ considerably from those of the NH4 H ats. A table is given showing the mol. extinction of various N-methylphenylacridonium salts, in different solvents. The ultraviolet absorption spectra have been determined of a large number of the compds. mentioned above, in a variety of solvents. The results are reproduced in the form of curves, in which the oscillation frequency is plotted against the thickness of the layer of liquid, in the usual manner. I. BISHOP TINGLE.

Hydromorphine. L. OLDENBERG. Inst. Inorg. Chem., Univ. Göttingen. Ber., 44, 1829-31.—Morphine hydrochloride, is reduced by Paal's method (C. A., 4, 314), in presence of Pd and H<sub>2</sub>O, to dihydromorphine, C<sub>17</sub>H<sub>21</sub>O<sub>2</sub>N.H<sub>2</sub>O; slender, colorless needles from MeOH, or alc., m. 155-7°. It resembles morphine in its bitter taste, in its reaction with FeCl<sub>2</sub> and in its narcotic effect and it reduces AgNO<sub>3</sub> instantly. Hydrochloride, C<sub>17</sub>H<sub>21</sub>O<sub>2</sub>N.HCl, from the base and HCl; mic oscopic prisms. Suljate, amorphous, from acetone.

J. BISHOP TINGLE.

Action of Phenol on Methylcoumaric Acid Dibromide and the Constitution of Werner's "Hydroxyphenylcoumaran." R. Stoermer and C. Friemel. Chem. Inst., Univ. Rostock. Ber., 44, 1838-53.—2-Methoxy-4'-hydroxystilbene, MeOC<sub>6</sub>H<sub>4</sub>CH: CHC<sub>6</sub>H<sub>4</sub>OH, Werner's "hydroxyphenylcoumaran," (Bcr., 39, 27 (1906)) is best prepared by m. on the H<sub>2</sub>O-bath a mixt. of equal pts. of PhOH and methylcoumaric acid dibromide, until the material ceases to be mobil; H<sub>2</sub>O is then added and subsequently Na<sub>2</sub>CO<sub>2</sub>; m. 149°, not 150-4°. In the prep. of methylcoumaric acid dibromide, for which detailed instructions are given, a pure product is obtained directly by the use of CS<sub>2</sub> instead of CHCl<sub>3</sub>, as recommended by Perkin (Ann., 216, 160 (1882)). 2-Methoxy-α-4'-hydroxyphenyl-β-bromohydrocoumaric acid, MeOC<sub>6</sub>H<sub>4</sub>CHBrCH(C<sub>6</sub>H<sub>4</sub>OH)CO<sub>2</sub>H. is obtained, before the Na<sub>2</sub>CO<sub>2</sub> is added; colorless, rhombic plates from warm glacial AcOH, m. 185-6°. Yield, 65%. The Br is eliminated by aq. NaOH (10%) and an acid is produced, but it could not be purified. It did not contain Br. 2-Methoxy-4'-ethoxy-stilbene, C<sub>17</sub>H<sub>18</sub>O<sub>2</sub>, from the above 4'-hydroxy compd. and Et<sub>2</sub>SO<sub>4</sub>; broad needles with a blue fluorescence from alc., m. 70°. 2-Methoxy-4'-benzoyloxystilbene, C<sub>12</sub>H<sub>16</sub>O<sub>3</sub>, by

the Schotten-Baumann method; colorless, silky lustrous needles from alc., m. 123°. a-Methoxy-4'-4"-nitrobenzoyloxystilbene, MeOC,H,CH: CHC,H,OCOC,H,NO, from pnitrobenzoyl chloride, in a similar manner to the preceding compd.; lustrous, golden yellow plates from glacial AcOH, m. 148°. 2,4'-Dimethoxystilbene dibromide, C<sub>14</sub>H<sub>14</sub>O<sub>2</sub>Br<sub>2</sub>, prepared in CS<sub>2</sub>; white crystals from Et<sub>2</sub>O + petroleum ether, m. 133°. 2,4'-Dimethoxystilbene is best reduced to the corresponding dibenzyl deriv. by means of Na and alc. 2-Methoxy-4'-hydroxydibenzyl, C14H10O2, is obtained if the stilbene be reduced by Na, in b. amyl alc., the solns. must be highly conc., so that the temp. will be high; long, colorless needles from ligroin, m. 63°. The stilbene is not reduced by Pt black and H (Paal). With Me,SO, the hydroxydibenzyl gives 2,4'-dimethoxydibenzyl (see above). 2-Methoxy-4'-benzoyloxydibenzyl, C2H20O2, colorless, silky lustrous needles from alc., m. 69°. p-Nitrobenzoyl derivative, is prepared like the preceding compd. from the acyl chloride and aq. NaOH; cream-colored plates, m. 135°. When oxidized by KMnO4, in presence of aq. NaOH, both the methoxyhydroxystilbene and dibenzyl give o-methoxybenzoic acid. Under similar conditions, 2,4'methoxystilbene forms a mixture of o- and p-methoxybenzoic acids. No condensation could be effected between o-methoxybenzyl cyanide and 4-methoxybenzaldehyde, but at 123-5°, in presence of Ac<sub>2</sub>O, the aldehyde and Na o-methoxyphenylacetate form a-2-methoxyphenyl-4'-methoxycinnamic acid, MeOC,H,CH: C(C,H,OMe)CO,H; almost colorless needles from alc., or glacial AcOH, m. 191°. Distillation with soda lime converts it into 2,4'-dimethoxystilbene. In a similar manner, o-methoxybenzaldehyde and Na p-hydroxyphenylacetate give α-4-hydroxyphenyl-2'-methoxycinnamic acid, MeOC, H, CH: C(C, H, OH) CO, H; aggregates of needles from alc., or glacial AcOH, m. 202°. Distillation with soda lime gives 2-methoxy-4-hydroxystilbene. When the acid is treated with Me,SO, and the product hydrolyzed a-4-methoxyphenyl-2'methoxycinnamic acid, MeOC,H,CH: C(C,H,OMe)CO,H, is produced, m. 198°. 2,4'-Dimethoxystilbene is obtained from it by distilling with soda lime. 2-Ethoxy-α-4'hydroxyphenyl-β-bromohydrocoumaric acid, EtOC<sub>2</sub>H<sub>2</sub>CHBrCH(C<sub>2</sub>H<sub>4</sub>OH)CO<sub>2</sub>H, is prepared from PhOH and ethylcoumaric acid dibromide; needles from glacial AcOH, m. 166°. It is converted by aq. Na, CO, into 2-ethoxy-4'-hydroxystilbene, EtOC,H,CH: CHC<sub>2</sub>H<sub>4</sub>OH; needles with a blue fluorescence from ligroin, m. 107°. It gives, with Me\_SO4 and alkali, 2-ethoxy-4'-methoxystilbene, EtOC4H4CH: CHC4H4OMe; opaque plates with a blue fluorescence from ligroin, m. 60. J. BISHOP TINGLE.

Phosphorus Tribromide as a Reducing Agent. III. Phenyl Migration in the Synthesis of Phenylated Coumarons. R. Stoermer. Chem. Inst., Univ. Rostock. Ber., 44, 1853-65; see C. A., 1, 2128.—2-Phenyl-4-methylcoumaron, formula (I) below, is prepared by heating phenyl-p-cresylacetolactone with PBr<sub>2</sub>, or P<sub>2</sub>S<sub>3</sub>; liquid, b<sub>20</sub> 193°. It has a sweet, aromatic odor and gives an intense red color with conc. H<sub>2</sub>SO<sub>4</sub>.

At 50-60°, Na and alc. reduce it to 2-phenyl-4-methylcoumaran. At 235-8°, during 18-20 hrs., PBr, converts the above lactone into *1-phenyl-4-methylcoumaron* (II), which was also obtained from 3-methyl-6-hydroxybenzaldehyde, phenylchloroacetic acid, NaOH, alc. and H<sub>2</sub>O, at 205-10°, during 15-18 hrs.; white, interlaced needles from dil. alc., m. 129°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is lemon-yellow, turning to green. 2-Phenylcoumaron and 1-bromo-2-phenylcoumaron are not changed by PBr<sub>2</sub>, at 235°. *1-Bromo-2-phenyl-4-methylcoumaron*, C<sub>12</sub>H<sub>11</sub>OBr, is formed from (I) and Br, in CS<sub>2</sub>; stellate needles from alc., m. 65°. With conc. H<sub>2</sub>SO<sub>4</sub> the color is pale red. The compd.

could not be prepared from phenyl-p-cresylbromoacetolactone. 1-Nitro-2-phenyl-4methylcoumaron, C11H11O2N, is prepared from the preceding compd. and HNO2 (as vapor), or NaNO, and conc. HNO, in glacial AcOH; aggregates of small, yellow needles, m. 115-6°. 1-Bromo-2-phenylcoumaron, C,4HeOBr, is formed in CS, oil with a sweet odor, b, 195°. It is reduced to 2-phenylcoumaron by Na and alc. 1-Nitro-2-phenylcommaron, C14HaO2N, from the Br compd. as above; long, yellow needles, m. 105°. 1-Phenylcoumaran, from hydroxydiphenylacetolactone and PBr2, at 235°, during 18-20 hrs., gives a bromide; silvery, lustrous plates from dil. alc., m. 81°. It does not react with HNO,. Phenyl-m-cresylacetolactone (III) and POCla, at 130°, during 15 hrs., form 1-chloro-2-phenyl-5-methylcoumaron, C1AH11OCl; silvery lustrous needles from dil. alc., m. 85.5°; b<sub>10</sub> 175°. Yield, 60%. It is converted by Na and alc. into 2-phenyl-5-methylcoumaran, C18H14O; small, white needles, m. 45°; b14 170°. Yield, good. With Br, in glacial AcOH, it gives 4-(?)-bromo-2-phenyl-5-methylcoumaran, C<sub>1.</sub>H<sub>12</sub>OBr; opaque needles, m. 125°. At 200°, during 18 hrs., alc. KOH converts it into unsym. phenyl-m-cresylethylene, HOC, H, MeCPh: CH2; almost colorless oil, b2, 205°. It dissolves in alkali and gives an intense red color with conc. H<sub>2</sub>SO<sub>4</sub>. 2-Phenyl-5-methylcoumaron (IV), is prepared from (III) by means of PBr<sub>2</sub>, or P<sub>2</sub>S<sub>1</sub>; opaque

needles with a sweet odor, m. 31°; b<sub>18</sub> 168°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is intensely orange-red, on warming it becomes lighter. Yield, 15 and 7.5% of (III), resp. On exposure to sunlight, in CS2, Br and (IV) form z-bromo-2-phenyl-5-methylcoumaron; m. 95°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is pale red. It gives, with AcOH, HNO<sub>2</sub> and NaNO<sub>2</sub>, I-nitro-2-phenyl-5-methylcoumoron; warty aggregates of small, yellow crystals from alc., m. 119-20°. The conversion of (III) into 1-phenyl-5-methylcournaron (V), by means of PBr, is attained only with considerable difficulty and the yield is very poor; small, silvery lustrous needles from alc., m. 135.5°. In conc. H,SO, the color is intensely yellow, changing gradually to dark green. The same compd. (V) was also prepared from 4-methyl-2-hydroxybenzaldehyde, phenylchloroacetic acid and alkali, at 200-5°. Yield, poor. 2-Hydroxy-4'-methoxydiphenylacetolactone, C1.H12O2, was obtained from p-methoxybenzaldehyde cyanohydrin, PhOH and H<sub>2</sub>SO<sub>4</sub> (73%); colorless needles from dil. alc., m. 178°. It could not be converted into a coumaron by treatment with PBr. or P.S. 2-Hydroxy-5-methyl-4'-methoxydiphenylacetolactone, C16H14O2, is produced in a similar manner from p-methoxybenzaldehyde cyanhydrin, p-cresol and H<sub>2</sub>SO<sub>4</sub> (73%); white plates, m. 135°. Yield, 60%. At 220°, during 12 hrs., PBr converts it into 2-[4'-methoxy]phenyl-4-methylcoumaron (VI); interlaced needles from dil. alc., m.

73.5°. In conc. H<sub>2</sub>SO<sub>4</sub> the color resembles that of eosin. Yield, 4%. It is still smaller by the use of P<sub>2</sub>S<sub>6</sub>. 2-Hydroxy-4'-chlorodiphenylacetolactone, C<sub>14</sub>H<sub>4</sub>O<sub>2</sub>Cl, is prepared from p-chlorophenol and mandelic acid; white needles, m. 125°. Yield, about 60%. 2-Hydroxy-4'-bromodiphenylacetolactone, from p-bromophenol, in a similar manner; white needles, m. 123°. Yield, about 60%. With POCl<sub>2</sub>, at 145°, during 9 hrs., the chloro lactone gives 1-chloro-2-[4'-chlorophenyl]coumaron (VII); long, silvery lustrous needles from alc., m. 122°. Yield, about 60%. It is reduced to 2-phenylcoumaran by Na and alc. At 200°, during 12 hrs., PBr<sub>2</sub> converts (VII) into 2-[4'-chloro]phenyl-

coumaron (VIII); crystallin, m. 34°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is brilliant orange. Yield, 30%; by the use of P<sub>2</sub>S<sub>2</sub>, instead of PBr<sub>2</sub>, the yield is 20%. r-Chloro-2-[4'-bromo]phenylcumaron, C<sub>14</sub>H<sub>2</sub>OClBr, is prepared from the bromolactone, in a similar manner to (VII); long, lustrous needles from dil. alc., m. 119°. Yield, about 60%. With PBr<sub>2</sub>, or P<sub>2</sub>S<sub>2</sub>, it gives 2-[4'-bromo]phenylcoumaron, vide (VIII); long, interlaced needles from dil. alc., m. 38°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is orange-red. Yield, about 20%. It was not possible to convert either this compd. or (VIII) into a 1-[4'-bromo-or chloro]phenylcoumaron by the action of PBr<sub>2</sub>, at a high temp. The author discusses the reactions involved in the migration of a Ph group, as described above, and assumes that from the lactones (IX), there are produced the intermediate complexes (X) from

which, by migration of either H or Ph, the isomeric commaron, such as (I) and (II), are formed.

J. BISHOP TINGLE.

By-products Obtained during the Preparation of Phloroglucinyldicarboxylic Ester. Hermann Leuchs and Fritz Sigion. Chem. Inst., Univ. Berlin. Ber., 44, 1874—84; see C. A., 3, 542.—The condensation of Et malonate is best accomplished by mixing the ester (200 g.) with abs. alc. (200 cc.) and Na (15 g.), distilling off the alc. and heating the residue at 135-40°, during 12 hrs. The product is sepd. by a somewhat complicated process, previously described. The compds. described below are obtained from the filtrate, after the removal of di-Et phloroglucinyldicarboxylate. Triethylester formula (I) below, is purified by soln. in cold petroleum ether, and subsequent

recrystn. from alc.; long, white, interlaced needles, m. 95.5-6.5°; mol. wt., in C<sub>2</sub>H<sub>4</sub>, 347-75. It gives a deep reddish brown color with FeCl<sub>3</sub> and a pale yellow soln. with cold, conc. HNO<sub>2</sub>. Yield, 3% of the reacting malonic ester. At 110-30°, KOH converts it into phloroglucinol. In alc., HONH<sub>2</sub> and (I) form a compound, C<sub>18</sub>H<sub>18</sub>O<sub>2</sub>N; small, lustrous, quadratic tablets, or prisms from C<sub>2</sub>H<sub>4</sub>, m. 203-4° (decomp.). In addition a more sol. substance is produced in small quantity; short needles from alc., m. about 230° (decomp.). Ac<sub>2</sub>O + AcONa convert (I) into the triacetyl compound (II); colorless prisms from alc., m. 109-10°. The mother liquor contains an ester, C<sub>18</sub>H<sub>19</sub>O<sub>13</sub>, in small quantity, it is apparently derived from the acid (m. 165° see below), some of which must have been present in the material employed; colorless needles, m. 145-6°. When b. with aq. HI (d. 1.7) (II) is hydrolyzed to the compound (III);

$$HOC \begin{picture}(200,0) \put(0,0){\line(1,0){100}} \put(0,0){\line(1,0$$

feathery aggregates of needles from glacial AcOH, not m. 300°. In presence of aq. "bicarbonate," it reduces "permanganate." Conc.  $H_2SO_4$  and (I) give an ester analydride,  $C_1$ ,  $H_{18}O_9$ ; long, hexagonal needles from  $C_0H_4$  and alc., m. 153-4°. Yield, 36% of (I). At the ordinary temp., I N aq. NaOH hydrolyzes (I) to the acid ester (IV); mas-

sive, prismatic crystals from  $C_0H_{40}$  m.  $162-3^{\circ}$ . At the ordinary temp., HNO<sub>2</sub> (d 1.4) and (I) form a compound,  $C_{14}H_{16}O_{40}$  which is also found among the original by-products, in that case too it is produced from (I) by the action of the acid employed; broad, colorless, lustrous needles from alc., m.  $128-9^{\circ}$ . In the acid mother liquor is a second compound,  $C_{16}H_{16}O_{10}$ ; needles, or prisms from alc., m.  $99-100^{\circ}$ . The original condensation product also contains an acid,  $C_{15}H_{16}O_{10}$ , which is doubtless formed by the action of alkali on its ester (see above); colorless, lustrous, acute-angled prisms from  $C_{9}H_{40}$  m. about  $165^{\circ}$  (gas evol.). When fused it evolves  $CO_{2}$  and gives the compd.  $C_{14}H_{16}O_{40}$  described above. With  $Ac_{2}O$  + AcONa the acid forms the ester,  $C_{20}H_{20}O_{19}$  (m.  $145-6^{\circ}$ ) described above. The original reaction product, after being heated at  $140^{\circ}$ , contains a compound,  $C_{20}H_{20}O_{19}$ ; pale yellow needles from alc., or glacial AcOH, m.  $196-7^{\circ}$ . In alc. it exhibits a feeble, greenish yellow fluorescence and it contains 3 EtO groups. The final by-product obtained was an ester (V); it is identical

$$EtO_{2}CCH \underbrace{\begin{array}{c} CO \cdot CH(CO_{2}Et) \cdot C \\ CO \end{array}}_{(V)} C \cdot CO \underbrace{\begin{array}{c} CHCO_{2}Et \\ CO \end{array}}_{(V)} C$$

with Willstätter's compd. (m. p. 177-8°) (Ber., 32, 1272 (1899)) and also with that to which O. Bally assigned the formula  $C_{12}H_{12}O_8$  (m. p. 170°) (Ber., 21, 1766 (1888)). At 100-30°, aq. KOH hydrolyzes it to phloroglucinol.

J. BISHOP TINGLE.

 $\Delta^{1.16}$ -Undecadiene and  $\Delta^{1.15}$ -Hexadecadiene. J. N. REFORMATSKII, E. GRISCH-KEVITSH-FROCHIMOVSKI AND A. SEMENZOV. Kiev. Ber., 44, 1885–6.— $\Delta^{1.10}$ -Undecadiene, CH<sub>2</sub>(CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH: CH<sub>2</sub>), is prepared by dissolving Mg in 1,5-dibromopentane and Et<sub>2</sub>O and adding allyl bromide; colorless, mobil liquid, b<sub>7M</sub> 187° (cor.); d<sup>20</sup> 0.7671; n<sup>20</sup> 1.43497; mol. ref. 51.73. Tetrabromo derivative, C<sub>11</sub>H<sub>20</sub>Br<sub>4</sub> formed in Et<sub>2</sub>O; colorless, viscid liquid. The hydrocarbon is oxidized by KMnO<sub>4</sub> to azelaic acid.  $\Delta^{1.15}$ -Hexadecadiene, CH<sub>2</sub>: CHCH<sub>2</sub>(CH<sub>2</sub>)<sub>16</sub>CH<sub>2</sub>CH: CH<sub>2</sub>, is formed together with the lower homolog and is separated by distillation; colorless, mobil liquid, or, at the temp. of snow and MeOH, cryst. solid, m. —14° to —12°; b<sub>4</sub> 142–7°; d<sup>18</sup> 0.8149; n<sup>18</sup> 1.45612; mol. ref. 74.07. Tetrabromo derivative, C<sub>16</sub>H<sub>20</sub>Br<sub>4</sub> pale red, viscid liquid. In petroleum ether, both hydrocarbons give light green, additive compounds with liquid N<sub>2</sub>O<sub>4</sub>. Yield, of C<sub>11</sub>H<sub>20</sub> and of C<sub>16</sub>H<sub>20</sub>, 18 and 9% of the dibromopentane, resp. J. B. T.

Acetohalogen Glucoses and Maltose and Melibiose p-Bromophenylosazones. Rmil Fischer. Chem. Inst., Univ. Berlin. Ber., 44, 1898–904; see C. A., 4, 3235.— $\alpha$ -Pentaacetylglucose and dry, liquid HCl give  $\beta$ -acetochloroglucose only. Earlier expts., carried out under the same conditions as nearly as possible, gave  $\alpha$ -acetochloroglucose, from which, by hydrolysis,  $\alpha$ -methylglucoside was obtained (Ber., 34, 2885 (1901)). With dry, liquid HBr,  $\alpha$ -pentaacetylglucose gave  $\beta$ -acetobromoglucose. The compd. described previously as " $\alpha$ -ace obromoglucose" is probably the slightly impure  $\beta$ -deriv. In glacial AcOH, dry HBr converts both  $\alpha$ - and  $\beta$ -pentacetylglucose into  $\beta$ -acetobromoglucose. Yield, 70 and about 88% of the  $\alpha$ - and  $\beta$ -compds., resp. The preparation of maltosone and of melibisone, according to the method described previously (Ber., 35, 3141 (1902)) is much facilitated by the presence of an organic acid, such as BzOH in the BzH, of AcOH, or phenylhydrazine acetate.  $\rho$ -Bromophenylmaltosazone and  $\rho$ -bromophenylmelibiosazone have been prepared once more by the method described previously and have been analyzed completely. Their properties and comp. are identical with those given in the earlier papers. J. B. T.

Compounds of the Propane Series. S. Gabriel. Lab, Univ. Berlin. Ber., 44, 1905-15.—Phthalimidobromoacetone, C<sub>2</sub>H<sub>4</sub>O<sub>2</sub>: NCH<sub>2</sub>COCH<sub>2</sub>Br, is prepared by mixing phthalimidoacetone and PBr<sub>2</sub>, the PBr<sub>3</sub> which is produced being distilled off under

reduced pressure; slender, colorless, interlaced needles from C<sub>2</sub>H<sub>2</sub>, m. 147-8°. The compd. is also formed, more conveniently, from phthalimidoacetone and Br, in CHCla. Yield, 85% of the imide, At about 170°, it reacts with K phthalimide giving diphthalimidoacetone, CO(CH<sub>2</sub>N:C<sub>2</sub>H<sub>4</sub>O<sub>2</sub>)<sub>2</sub>. Dry AcONa and the bromoacetone, at 170°. form phthalimidohydroxyacetone, CaHaOa: NCHaCOCHaOH; silky lustrous needles from alc. (96%), m. 141-2°. It is also produced, in small quantity, together with phthalimide and methylglyoxal, AcCHO, (isolated and identified as the phenylosazone) by b. the bromoscetone compd. with H.O. during 5 hrs. Phthalimidodibromoacetone, CaHaOa: NCHBrCOCHaBr, is prepared from phthalimidoacetone and Br, in warm CaHa; long needles resembling KNOa from CaHa, m. 126-7°. It is hydrolyzed by b. H<sub>2</sub>O to phthalimide and bromomethylglyoxal, BrCH<sub>2</sub>COCHO, which was isolated as bromomethylglyoxalphenylhydrazone, BrCH\_COCH: NNHPh; mahogany-colored powder consisting of oblong plates and needles from alc. containing a little AcOH. m. 164-6° (gas evolution). At 50-60°, phthalimidoacetone and Br, in C.H., give phthalimidotribromoacetone, CaH4O3: NCHBrCOCHBr3; acute-angled rods from CaH4 m. 146-7°. It is hydrolyzed by b. H.O to phthalimide and dibromomethylglyoxal which was isolated as dibromomethylglyoxalphenylhydrazone, Br.CHCOCH: NNHPh; lemon-yellow needles from alc., m. 174-5°. When warmed with Br the tribromo compd. is converted into phthalimidotetrabromoacetone, C<sub>2</sub>H<sub>4</sub>O<sub>2</sub>: NCHBrCOCBr<sub>2</sub> rhombic plates from C<sub>2</sub>H<sub>4</sub>, m. 153-4°. It is prepared more conveniently by adding Br to well cooled phthalimide and subsequently heating the mixture at 80°. Yield, 56% of the imide. B. with H<sub>2</sub>O hydrolyzes it to phthalimide, CHBr<sub>2</sub>, HBr and probably HCHO. B., dil. alkali causes an immediate production of CHBr.. Propenylphthalimide, C<sub>2</sub>H<sub>4</sub>O<sub>2</sub>: NCH: CHMe, is obtained by distilling phthalimidobutyryl chloride in a vacuum. Yield, 40% of the chloride. Phthalimidotrimethylene,

C<sub>0</sub>H<sub>4</sub>CONCHCH<sub>2</sub>, is prepared by evaporating a mixt. of equimol. solns. of CH<sub>2</sub>

phthalic acid and aminotrimethylene and distilling the residue in a vacuum; rhombic plates from alc., m. 135–6°. In CHCl<sub>2</sub>, Br and propenylphthalimide form phthalimido- $\alpha$ , $\beta$ -dibromopropane, C<sub>2</sub>H<sub>4</sub>O<sub>3</sub>: NCHBrCHBrMe; flat, hexagonal crystals from ligroin, m. 105–6°. It is not identical with either phthalimido- $\beta$ , $\gamma$ -dibromopropane, C<sub>2</sub>H<sub>4</sub>O<sub>3</sub>: NCH<sub>2</sub>CHBrCH<sub>2</sub>Br, (m. 113–4°), nor with Hildesheimer's phthalimidodibromopropane (m. 147°) from phthalimidobutyric acid, Br and P (C. A., 5, 486), consequently this latter cannot be the  $\alpha$ , $\beta$ -compd. as above, but must have the  $\alpha$ , $\gamma$ -formula, C<sub>2</sub>H<sub>4</sub>O<sub>2</sub>: NCHBrCH<sub>2</sub>CH<sub>2</sub>Br. The  $\alpha$ , $\beta$ -compd. is hydrolyzed by b. with H<sub>2</sub>O, during 4 hrs., to phthalimide and acetol, which was isolated as acetolphenylhydrazone, HOCHMeCH: NNHPh. Phthalimido- $\alpha$ , $\gamma$ -dibromopropane has been stated to give these same hydrolysis products.

Grignard's Reaction and its Application to Dihalogen Compounds. I. J. v. Braun and W. Sobrcki. Chem. Inst., Univ. Breslau. Ber., 44, 1918-31.—Mg and 1,4-dibromobutane were allowed to react and the product treated with CO<sub>2</sub>, the substance so formed were sepd. by means of aq. alkali. They consisted of cyclopentanone, C<sub>4</sub>H<sub>6</sub>CO, and sebacic and dodecamethylenedicarboxylic acids, the latter of which is more sol. in Et<sub>2</sub>O. No trace of butylene could be detected. The reaction product of Mg and 1,5-dibromopentane, when treated with H<sub>2</sub>O, gave pentane, decane pentadecane and eicosane, together with higher paraffins. Yields, 35, about 15, less than 10, a few % and about 15%, resp. of the dibromo compd. No cyclopentane was formed. Under similar conditions, 1,7-dibromoheptane and Mg produce heptane, tetradecane and higher b. products. No trace of cycloheptane could be detected. Treatment of the Mg derivative with CO<sub>2</sub> gives, chiefly, heptamethylenedicarboxylic

acid. 1,10-Diiododecane reacts even more slowly with Mg than does 1,7-dibromoheptane, the products, after treatment with H<sub>2</sub>O, are decane, eicosane and, possibly, tetracontane, C<sub>40</sub>H<sub>42</sub>. Decamethylenedicarboxylic acid is the chief substance formed by the interaction of CO<sub>2</sub> and the preceding Mg derivative. In addition to the glycol, (HOCMe<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>, described previously, Mg, 1,5-dibromopentane and acetone give the alc., HOCMe<sub>2</sub>CH<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>Me, and the alcohol, HOCMe<sub>2</sub>CH<sub>2</sub>(CH<sub>2</sub>)<sub>4</sub>CH<sub>2</sub>CMe: CH<sub>2</sub>, or HOCMe<sub>2</sub>CH<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>CH: CMe<sub>2</sub>, it may, however, be a mixture of both; liquid, b<sub>14</sub> 107-9°; d<sup>20</sup>/<sub>4</sub> 0.8467°; n<sub>D</sub> 1.45512; mol. ref. 54.49. The substance has a pleasant odor of citronellol. The above compd. from dibromopentane are sepd. by distillation. The glycol mentioned above crystallizes from Et<sub>2</sub>O + ligroin, m. 77°; b<sub>12</sub> 135-54°. With anhydrous HBr, in glacial AcOH, it forms a dibromide, C<sub>11</sub>H<sub>22</sub>Br<sub>3</sub>; viscid oil. When b. with pyridine it gives a mixture of unsaturated hydrocarbons, which could not be separated.

Spectrographic Studies in the Phthalein Group. RICHARD MEYER AND OTTO FISCHER. Chem. Lab., Techn. Hochsch., Braunschweig. Ber., 44, 1944-54.—The authors have measured the ultraviolet absorption spectra of the following compds.: phenolphthalein, fluorescein, hydroxydiphenylphthalide, tetrabromophenolphthalein, hydroquinolphthalein and hydroxyphenylphthalide. The results are given in tabular form and are also reproduced as curves. The 2 phthalides give spectra which are markedly different from those of the true phthaleins. No general conclusions are drawn. The following derivs. of hydroxyphenylphthalide, formula (I) below (R = H),

$$C_{\bullet}H_{\bullet} \underbrace{\begin{array}{c} -CHC_{\bullet}H_{\bullet}OR \\ | \\ CO \cdot O \\ (I) \end{array}}_{CO \cdot NPh} C_{\bullet}H_{\bullet} \underbrace{\begin{array}{c} -CHC_{\bullet}H_{\bullet}OH \\ | \\ CO \cdot NPh \\ (II) \end{array}}_{CO \cdot NPh}$$

have been prepared. Ethyl ether (R = Et), from EtI and EtOK; rhombic plates from alc.  $+ H_2O$ , or AcOH  $+ H_2O$ , m. 116°. In conc.  $H_2SO_4$  the color is yellow. Benzoyl derivative (R = Bz), by the Schotten-Baumann method; colorless, rectangular plates from glacial AcOH, m. 188°. In conc.  $H_2SO_4$  the color is pale yellow. Benzyl ether ( $R = CH_2Ph$ ), from benzyl chloride and alc. NaOH; prismatic needles from glacial AcOH, m. 180°. In conc.  $H_2SO_4$  the color is yellow. Antiide (II), from the phthalide, PhNH<sub>2</sub>-HCl and b. PhNH<sub>2</sub>; colorless needles from glacial AcOH  $+ H_2O_4$  darkens 250°, m. 267°. In conc.  $H_2SO_4$  the color is yellow. The following derivator hydroxydiphenylphthalide (III) (R' = H) have been prepared by the methods used for (I). Benzoyl derivative (R' = Bz), triangular prisms from glacial AcOH,

$$C_{\mathfrak{g}}H_{\mathfrak{q}} \underbrace{\begin{array}{c} -CPhC_{\mathfrak{g}}H_{\mathfrak{q}}OR' \\ CO \cdot O \\ (III) \end{array}}_{C\mathfrak{g}}C_{\mathfrak{g}}H_{\mathfrak{q}} \underbrace{\begin{array}{c} -CPhC_{\mathfrak{g}}H_{\mathfrak{q}}OH \\ CO \cdot NPh \\ (IV) \end{array}}_{C\mathfrak{g}}$$

m. 139°. In conc.  $H_2SO_4$  the color is orange. Ethyl eth r (R' = Et), quadratic tablets from ligroin, m. 88°. In conc.  $H_2SO_4$  the color is orange. Methyl ether (R' = Me), from  $Me_2SO_4$  and 1 N aq. KOH; rectangular prisms from glacial AcOH, m. 86°. In conc.  $H_2SO_4$  the color is reddish yellow. Benzyl ether ( $R' = CH_2Ph$ ), rectangular prisms from glacial AcOH, m. 109°. In conc.  $H_2SO_4$  the color is orange. Anilide (IV), hexagonal plates from glacial AcOH, m. 276°. In conc.  $H_2SO_4$  the color is intensely yellow.

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Composition of Phthalein Salts. RICHARD MEYER AND FERD. POSNER. Chem. Lab., Techn. Hochsch., Braunschweig. Ber., 44, 1954-7.—The red Na salt of phenolphthalein and the violet deriv. of hydroquinolphthalein have the formulas C<sub>20</sub>H<sub>12</sub>O<sub>4</sub>Na<sub>2</sub> and C<sub>20</sub>H<sub>10</sub>O<sub>4</sub>Na<sub>2</sub>, resp. This confirms the author's previous results, but it does not agree with Kehrmann and v. Baeyer's formulas, C<sub>20</sub>H<sub>11</sub>O<sub>4</sub>Na<sub>2</sub>, and C<sub>20</sub>H<sub>10</sub>O<sub>4</sub>Na<sub>4</sub>, resp.,

for the hydroquinol compd. (C. A., 4, 2124, 2130). The following precautions must be observed in the prep. and analysis of the above Na salts. The aq. NaOH which is employed must be free from Na<sub>2</sub>CO<sub>2</sub> and the undissolved phthalein must be filtered as quickly as possible, in order to avoid pptn. of phthalein by the CO<sub>2</sub> in the air. Excess of acid must not be used to decomp. the Na salt and the pptd. phthalein must be allowed to remain over night, for crystn. The phthalein remaining dissolved in the neutralized liquid must be recovered and weighed. Calcium phenolphthalein, C<sub>20</sub>H<sub>12</sub>O<sub>4</sub>Ca.2.5H<sub>2</sub>O, is prepared by mixing finely divided Ca(OH)<sub>2</sub> and phenolphthalein with H<sub>2</sub>O; after shaking, the liquid is filtered and the filtrate b. to ppt. the salt, care being taken to avoid exposure to CO<sub>2</sub>. Reddish brown, flat prisms with a green metallic luster; when powdered it is dark red. The salt is stable in air; when anhydrous it is green and it passes into the hydrated salt on exposure to moisture. This salt does not appear to be identical with that described in Ger. Pat. 223,969 (C. A., 4, 3282).

J. BISHOP TINGLE.

Naphthoresorcinol [1,3-Dihydroxynaphthalene] and 1-Amino-3-naphthol. RICHARD MEYER AND KURT WOLFSLEBEN. Chem. Lab., Techn. Hochsch., Braunschweig. Ber., 44, 1958-66.—1,3-Dihydroxy-2,4-dibromonaphthalene, C<sub>10</sub>H<sub>4</sub>Br<sub>2</sub>(OH)<sub>2</sub>, is prepared by adding Br slowly to the dihydroxynaphthalene, in glacial AcOH, at 0°; colorless needles from AcOH + H<sub>2</sub>O, m. 128-9°. It is unstable in air. With glacial AcOH, Ac<sub>2</sub>O and a little conc. H<sub>2</sub>SO<sub>4</sub>, at the ordinary temp., it gives a diacetyl derivative, C<sub>10</sub>H<sub>4</sub>Br<sub>2</sub>(OAc)<sub>2</sub>; colorless crystals from MeOH, m. 125°. Under similar conditions to those described above, but with more Br, the dihydroxynaphthalene forms tribromo-1,3-dihydroxynaphthalene, C<sub>10</sub>H<sub>2</sub>Br<sub>2</sub>(OH)<sub>2</sub>; silvery lustrous needles from CS<sub>2</sub>, m. 186° (decomp.). Diacetyl derivative, by b. with Ac<sub>2</sub>O + H<sub>2</sub>SO<sub>4</sub>; monoclinic needles and prisms fron glacial AcOH, m. 182°. Dibensoyl derivative, from B2Cl, at 150°; interlaced needles from acetone, m. 129°. At 0°, in H<sub>2</sub>O, Br and 1,3-dihydroxynaphthalene give a naphthalenehetotribromide formula (I), or (II) below; yellow crystals

$$OH \\ Br \\ = O \\ Br_{t}$$

$$(I) \\ Br_{t}$$

$$(II)$$

from C<sub>2</sub>H<sub>2</sub> + ligroin, decomp. 85°. With AcCl it gives the above 1,3-diacetoxy-2,4dibromonaphthalene. The 2,4-position assigned above to the Br ats. is essentially hypothetical. Hydroxynaphthoquinone (m. 190°) was formed by passing air, free from CO, thru an alkaline soln. of 1,3-dihydroxynaphthalene, during 6 hrs. Dibenzoyl-1-amino-3-naphthol, C10He(NHBz)OBz, is prepared from the aminonaphthol hydrochloride and BzCl, at 130°; colorless needles from glacial AcOH, m. 188°. Ethyl 3-hydroxynaphthyloxamate, HOC10HaNHCOCO1Et, is obtained by b. 1-amino-3-naphthol with di-Et oxalate; pale yellow, interlaced needles from CHCla, m. 171°. It is hydrolyzed to the acid by b. dil., aq. NaOH; needles from MeOH, m. 219° (decomp.). Amide, HOC, HANHCOCONH, from the ester and hot, aq. NH, followed by HCl; flat, yellow needles from alc., m. 260°. 3-Hydroxynaphthyl-I-phthalamic acid, HOC10HaNHCOC4HaCO4H, is prepared by mixing the aminonaphthol with phthalic anhydride, in hot xylene; yellow crystals from dil. alc., m. 220° (decomp.). Yield, poor. In the same manner were prepared  $\alpha$ - and  $\beta$ -naphthylphthalamic acids, C, H,NHCOC,H,CO,H. I-Benzoylamino-3-benzoyloxybromonaphthalene, (NHBz)OBz, is formed from the above di-Bz compd. and Br, in alc.; colorless crystals. I-Acstaminodibromo-3-naphthol, HOC<sub>10</sub>H<sub>4</sub>Br<sub>2</sub>NHAc, is prepared from the Ac deriv. and Br, in glacial AcOH; thick needles from glacial AcOH, m. 210° (decomp.). No proof is offered of the position of the Br ats. in the preceding 2 compds. Diacetyldibromo-1-amino-3-naphthol, C<sub>10</sub>H<sub>4</sub>Br<sub>2</sub>(NHAc)OAc, is obtained from the preceding compd., Ac<sub>2</sub>O and conc. H<sub>2</sub>SO<sub>4</sub> and also by brominating 1-amino-3-naphthol, in alc., and b. the resulting product with glacial AcOH and Ac<sub>2</sub>O; colorless needles from glacial AcOH, m. 230°.

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o,o'-Azoxybenzaldehyde. Eug. Bamberger. Anal. Chem. Lab., Federal Polytech., Zurich. Ber., 44, 1966-79.—o,o'-Azoxybenzaldehyde diethylacetal, formula (I) below, is prepared by b. o-nitrobenzaldehyde diethylacetal, MeOH and MeONa, or NaOH, during 4 hrs.; with 30 g. of the acetal, the b. continued during 8-10 hrs. Colorless prisms from alc., m. 76.5°. Yield, 83% of the nitro compd.; by the use of NaOH the yield is smaller. o,o'-Azoxybenzaldehyde dimethylacetal was prepared in a similar manner from the nitro methylacetal; colorless, rhombic plates from petroleum ether, m. 58.5-9.5°. In glacial AcOH, dil. aq., HCl hydrolyzes (I), at the ordinary

$$(EtO)_{1}CHC_{6}H_{4}N \cdot NC_{6}H_{4}CH(OEt)_{2} \qquad C_{6}H_{4} \stackrel{N}{\underset{C(OH)}{\bigcap}} NC_{6}H_{4}CO_{2}H$$

temp., to o,o'-azoxybenzaldehyde. Yield, 88%. At 85°, aq NaOH converts the a dehyde into indazolylbenzoic acid (II), which is also formed, together with indazolylbenzoic lactone (III) and indazylbenzoic acid (IV), by treating the aldehyde with

$$C_{\bullet}H_{\bullet} \swarrow \bigvee_{C} NC_{\bullet}H_{\bullet} \qquad \qquad C_{\bullet}H_{\bullet} \swarrow \bigvee_{CH} NC_{\bullet}H_{\bullet}CO_{\bullet}H$$

$$(IV)$$

glacial AcOH, at a temp. not exceeding 70°. The liquid is cooled and filtered; (IV) is contained in the filtrate and (II) and (III) are sepd. by means of aq. NaOH. o-Nitrobenzylanthranilic acid, O<sub>2</sub>NC<sub>2</sub>H<sub>4</sub>CH<sub>2</sub>NHC<sub>2</sub>H<sub>4</sub>CO<sub>2</sub>H, is reduced to (IV) by b. with alc., Sn and a little conc. HCl. o,o'-Azoxybenzaldehyde is not changed after about 3 yrs. in darkness. Insolation in ligroin, from Nov. 28, 1905 to Jan. 9, 1906, during which period there were only 6-7 fully clear ds., converted the aldehyde in part, into (III), together with a little of (II), apparently. Some amorphous material is also formed.

J. BISHOP TINGLE.

Hydrogenation and Dehydrogenation by Catalysis. PAUL SABATIER. Ber., 44, 1984-2001.—A lecture.

J. BISHOP TINGLE.

Refutation of E. Biilmann's Identification of Homochromisomerism as Polymorphism. A. Hantzsch. Ber., 44, 2001-9.—In reply to Biilmann (C. A., 5, 2249), who attacked his conception of homochromoisomerism (C. A., 4, 2648), the author points out that his results in connection with the 2 forms of methylphenylpicramide are identical with those of Biilmann. An apparent exception refers to the transformation of the lower into the higher m. form, by recrystn. from C<sub>6</sub>H<sub>6</sub>. This is due to a clerical error, the statement should have been that the C<sub>6</sub>H<sub>6</sub> was evapd. For convenience the 2 forms of the amide, m. 108° and 128°, will be referred to as A and B, resp. They are mutually transformable by heating, or recrystn. from specific media (Hantzsch and Lister) and also by inoculation (Biilmann). Each form may, however, also be recryst, without change, each is monomol, in soln, and each may be pptd., in its original form, from highly dil, solns. These results exclude polymorphism, but if it be stated that momomol, solns, of 2 polymorphic substances exist, then they must behave alike

under identical conditions. This, however, is not the case with the compds. in question, for each form, A and B, is deposited from its resp. soln. In the absence of catalysts, the differences must be intra- and not intermol., i. e., the compds. must be isomeric. The transformation of B into A, in soln. and of A into B by fusion, are time phenomena, the velocity rizing with the temp. This behavior, characterizes true chem. reactions. At 18°, the solubility factor g./cc., in alc., of A is 0.0032 and of B 0.0042. The results of inoculation expts. have but little value in deciding such cases as that in question, because the effects which are produced are due to catalysis. As the lab. and the clothes of the workers become "infected" with dust, it is increasingly difficult to prepare both A and B with certainty, the former is apt to be the only product. As the labil cinnamic acids have been shown to be optically identical, it is probable that they also are homochromoisomers. There is ground for regarding polymorphism as a sort of "quarantine" for the reception of compds., before their admission as isomers.

Constituents of Ethereal Oils. I. Identity with Myrcene of the Aliphatic Terpene from Oil of Hops. II. Methyl Esters of Dicarboxylic Acids. III. Preparation of Isobornyl Formate. F. W. Semeler and Erwin W. Mayer. Techn. Hochsch., Breslau. Ber., 44, 2009–12.—The aliphatic terpene in oil of hops was proved to be identical with myrcene by its own properties and by the preparation of dihydromyrcene, its tetrabromide and of myrcenol. Di-Me isofenchonecarboxylate,  $C_{12}H_{20}O_4$ ,  $b_{14}$  131–2°;  $d_{20}$  1.0515;  $n_D$  1.4540;  $\alpha_D$  —19°; mol. ref. 58.72. Di-Me camphorate,  $b_{14}$  137–9°;  $d_{20}^{20}$  1.0747;  $n_D$  1.4633. Brühl and Braunschweig give  $b_{11}$  149.5°;  $b_{11}$  155° (Ber., 25, 1809 (1892)). Di-Me  $\alpha$ -tanacetogendicarboxylate,  $b_{13}$  129–30°;  $d_{20}^{20}$  1.0525;  $n_D$  1.4510;  $\alpha_D$  148°. Di-Me  $\beta$ -methyladipate,  $b_{13}$  122–2.5°;  $d_{20}$  1.0372;  $n_D$  1.4336. The above esters were prepared from the Ag salts and MeI. Isobornyl formate,  $C_{11}H_{20}O_{20}$  is prepared by b. camphene with HCO<sub>2</sub>H (2 pts.). Yield, 120% of the camphene. When hydrolyzed the ester gives isoborneol, together with a little borneol.

J. BISHOP TINGLE.

The Two Stereoisomeric 1,2-Diphenyl-2-nitro-1-methoxyethanes Formed by the Addition of an Alkali Methylate to 7-Nitrostilbene. FRIEDRICH HEIM. Chem. Lab., Agric. Acad., Bonn-Poppelsdorf. Ber., 44, 2013-6; see J. Meisenheimer, C. A., 2, 78.—The 2 stereoisomeric forms of 1,2-diphenyl-1-methoxy-2-nitroethanes are best prepared by dissolving 7-nitrostilbene (22.5 g.) in MeOH (100 cc.) and adding to the cooled liquid, containing finely divided solid, a soln. (46 cc.) of Na (5 g.) in MeOH (100 cc.). From this soln. purified CO<sub>2</sub>, or NH<sub>4</sub>Cl and air ppt. the largest proportion of the lower m.,  $\beta$ -isomer, (7.61 and 6.03 g. resp.) whereas a low temp. favors the production of the higher m., a-form (18.58 and 19.70 g., resp.). The isomers are best separated mechanically, after recrystallization from petroleum ether. 7-Nitrostilbene is regenerated if the temp, is raised unduly during the pptn, as described above. The  $\alpha$ - is converted, in part, into the  $\beta$ -form by treatment with MeONa as above and pptn. with NH<sub>4</sub>Cl + air, at 50-60°. Yield, 17.61 g. of  $\alpha$ - and 5.91 g. of  $\beta$ -, from 25.07 g. of  $\alpha$ -. After 2.5 hrs. at 170°, 90% of the  $\beta$ -form (m. 97–8°) was recovered, only a trace of 7-nitrostilbene being produced. Under similar conditions, after only 0.5 hr., 63% of α-compd. (m. 130-1°) was recovered, together with 6% of nitrostilbene (m. 75°) and a trace of the isomer (m. 75°). This treatment failed to cause a transformation of either  $\alpha$ - or  $\beta$ -form into the isomeric modification (see following abstr.).

J. BISHOP TINGLE.

Condensation of Phenyinitromethane with Benzaldehyde. Cis- and Trans-7-Nitrostilbene. FRIEDRICH HEIM. Ber., 44, 2016-22; see preceding abstr.—The yield of 7-nitrostilbene, prepared by Knoevenagel and Walter's method (Ber., 37, 4509), was only 50% instead of 60-70% and the product did not m. sharply. Recrystalliza-

tion from petroleum ether separated it into 2 isomers, the ordinary, long, pale yellow needles (m.  $75^{\circ}$ ) and opaque, dark yellow, or greenish crystals, m.  $128-8.5^{\circ}$ . Yield, 7-10%. Both isomers give a mixture of  $\alpha$ - and  $\beta$ - 1,2-diphenyl-2-nitro-1-methoxymethane by the action of MeONa. The 7-nitrostilbenes are mutually transformable when heated or distilled. The alc. mother liquor, obtained during the preparation of the preceding compds., contains 3 other substances. Yield, 3.5-5% of each. After prolonged standing the alc. deposits the first compd. which is probably 7'-benzyl-7-nitrostilbene [1,2,3-triphenyl-1-nitro-1-propylene], PhCH<sub>2</sub>CPh: CPhNO<sub>2</sub>; yellow, interlaced crystals from alc., slender needles from petroleum ether, m.  $102-3^{\circ}$ . It reduces "permanganate" and adds alkali methoxides, forming a white substance. The other 2 compds. are separated by distilling the original alc. filtrate from the above propylene derivative. 1,2,3-Triphenyl-1,3-dinitropropane,  $(O_2$ NCHPh)<sub>2</sub>CHPh; slender, white, interlaced needles, m.  $177-8.5^{\circ}$ . It may be pptd. by adding alc. to the filtrate from the above propylene deriv. Conc., aq. alkali decomp. it into HNO<sub>2</sub> and  $\alpha,\beta,\gamma$ -tri-

phenylisoxazole, PhC | , which is also present in the original mother liquor CPh : N

and may be prepared by treating 7-nitrostilbene with excess of conc. aq. KOH (1:1), on a b.  $H_2O$ -bath: cryst., m. 212°. It is not changed by prolonged b. with conc. alkali, or conc. HCl. The compd. appears to be identical with that from aq. KOH and sym.  $\beta$ -diphenyldinitroethane, to which J. Schmidt gave the impossible formula  $C_mH_{10}ON$  (Ber., 34, 3542). In Richter's "Lexikon" the formula is given as  $C_mH_{17}ON$ , which is also incorrect. The relationship of the above compds. is discussed. No statement appears to be made as to which of the isomeric 7-nitrostilbenes is the cis- and which is the trans- form.

J. BISHOP TINGLE.

New Type of Anomalous Rotatory Dispersion. Optical Superposition. L. CHUGAEV. Chem. Lab., Imp. Univ., St. Petersburg. Ber., 44, 2023-30.—The author discusses the 2 known types of anomalous rotatory dispersion and also optical superposition. l-Menthyl  $d_{i}\beta$ -camphorsulf nate,  $C_{10}H_{18}OSO_{2}OC_{10}H_{18}$ , is prepared from the sulfonyl chloride and l-menthol, in presence of pyridine, or from Na mentholate; crystallin. It gives the following values for  $\alpha$ ; at 20°, for the lines and solvents mentioned: C 9.76, in toluene, C—12.04°, D—13.45°, E—13.95°, F—12.86°,  $\lambda = 472 \mu\mu$ —12.04°. C 7.54, in acetone, C -15.38°, D -17.57°, E -19.08°, F -18.73°,  $\lambda = 472 \mu\mu$ --18.03°. C--9.66, in CHCl<sub>2</sub>, C--17.87°, D--20.63°, E--22.92°, F--23.30°, λ=472 μμ  $-22.76^{\circ}$ ,  $\lambda = 457 \,\mu\mu$   $-21.46^{\circ}$ . l-Menthyl  $l_{i}\beta$ -camphorsulfonate gives the following values, under similar conditions: C 9.70. in toluene, C -57.9°, D -74.7°, E -98.3°, F -122.0°,  $\lambda = 472 \mu\mu$ , -131.3° [\alpha]F/[\alpha]C 2.11. C 11.05, in acetone, C -57.3°, D  $-74.1^{\circ}$ , E  $-97.8^{\circ}$ , F  $-121.4^{\circ}$  [ $\alpha$ ]F/[ $\alpha$ ]C 2.12. These results show that, in the case of both esters, neither the abs. refractive power, nor the dispersion is materially affected by the solvent. The *l*-menthol  $d_i\beta$ -camphorsulfonate apparently affords an instance of intramol. anomalous dispersion. J. BISHOP TINGLE.

Position of the Substituents in Homohydroxysalicylic Acid. Hans Schmid. Chem. Lab., Imp. and Roy. Univ., Innsbruck. Monatsh., 32, 435-45.— Acetyltoluhydroquinol was prepared from the phenol, Ac<sub>2</sub>O and aq. KOH; long, lustrous needles from petroleum ether, m. 92°. It could not be oxidized to a carboxylic acid. Diacetyltoluhydroquinol is prepared by b. the phenol with Ac<sub>2</sub>O; with CrO<sub>2</sub> it gives only a resin, but aq. KMnO<sub>4</sub> + MgSO<sub>4</sub> oxidize it to hydroquinolcarboxylic acid, m. 196-7°. Homohydroxysalicylic acid was prepared from toluhydroquinol, KHCO<sub>2</sub> (2 pts.) and glycerol (2 pts.), at 180°, during 24-30 hrs., in an atm. of CO<sub>2</sub>. Yield, 40-5%. Diacetyl derivative, C<sub>2</sub>H<sub>4</sub>O<sub>4</sub>Ac<sub>2</sub>, from the acid and b. Ac<sub>2</sub>O; crystals from H<sub>2</sub>O, m. 129°. Yield, 60-70%. It is oxidized to 2,5-dihydroxyterephthalic acid by KMnO<sub>4</sub> + MgSO<sub>4</sub>.

Yield, 10-5%. The yield is still smaller by the use of KMnO<sub>4</sub> and anhydrous acetone. These result show that "homohydroxysalicylic acid," the "toluhydroquinolcarboxylic acid No. 25" (Beilstein, "Handbuch," Suppl. Vol. 2, p. 1033) and the acids Nos. 21 and 24 (M. M. Richter, "Lexikon d. Kohlenstoffver," 3 ed. p. 797) must all be 4-methyl-2,5-dihydroxybenzoic acid.

J. BISHOP TINGLE.

Formation of Flavanthrene. ERWIN BENESCH. Chem. Lab., Univ. Graz. Monatsh., 32, 447-56.—2-Methoxyanthraquinone, formula (I) below, when b. during 2 hrs., with HNO<sub>3</sub> (d. 1.37), gives 2 nitro compds., which are sepd. by means of glacial AcOH. 1-Nitro-2-methoxyanthraquinone (II), is the more sparingly sol.; yellow crystals

$$(I) \qquad (II) \qquad (III) \qquad (III)$$

from PhNO, m. 271°. It constitutes 60% of the total yield. 3-Nitro-2-methoxyanthraquinone (III), is formed together with (II); yellow crystals from glacial AcOH, m. 225°. Both (II) and (III) are reduced to the resp. amino derivs, by the action of aq. Na.S. 1-Amino-2-methoxyanthraquinone, from (II); brick-red crystals from glacial AcOH, m. 224°. 3-Amino-2-methoxyanthraquinone, from (III); m. 218-22°. The constitution assigned to this compd. and to (III) appears to be wholly hypothetical and that given to (II) and its derivs. is largely so. 1-Iodo-2-methoxyanthraquinone, is prepared by diazotizing the amines; brown, lustrous crystals from glacial AcOH, or alc., m. 265°. 3-lodo-2-methoxyanthraquinone is obtained in a similar manner to the preceding compd., which it resembles; m. 210-2°. 2,2'-Dimethoxy-1,1'-dianthraquinonyl (IV), is prepared by heating the 1-iodo compd. with Cu powder, at 360, during 10 mins., the product being extracted with C.H., at the ordinary temp. The residue is recrystallized from xylene, then from glacial AcOH; yellow powder, m. 346°. Yield, 20%. 2,2'-Dimethoxy-3,3'-dianthraquinonyl is prepared in a similar manner from the 3-iodo compd. Yield, about 2%. At 145°, during 0.5 hr., finely divided AlCla hydrolyzes (IV) to 2,2'-dihydroxy-1,1'-dianthraquinonyl, which was purified by dissolving

in aq. NH<sub>2</sub> and ppt. with acids; green, amorphous, very sparingly sol. powder. Yield about 90%. At 270°, during 24 hrs., conc., aq. NH<sub>2</sub> converts the preceding compd. and also (IV) into flavanthrene (V). Yield, extremely small. Under similar conditions, 2,2'-dimethoxy-3,3'-dianthraquinonyl fails to give even a trace of (V).

J. BISHOP TINGLE.

3,4,5-Trinitroveratrole. ALFONS KLEMENC. I. Chem. Lab., Imp. and Roy. Univ., Vienna. *Monatsh.*, 32, 457-9.—3,4,5-Trinitroveratrole is a product of the interaction of fuming HNO<sub>3</sub> and hemipinic acid, without a solvent. It is separated

by its insol. in aq.  $NH_2$ . The trinitro compd. is also formed by heating 5,6-dinitro-2,3-dimethoxybenzoic acid with  $HNO_4$  (d. 1.52), during 1 hr., on a b.  $H_2O$ -bath.

J. BISHOP TINGLE.

Anthraquinone Series. V. Dichloroanthraquinones. FRITZ ULLMANN AND GER-HARD BILLIG. Techn. Chem. Inst., Roy. Techn. Hochsch., Berlin. Ann., 381, 11-28; see C. A., 5, 3245.—3,6-Dichloro-2-benzoylbenzoic acid, Cl<sub>2</sub>C<sub>4</sub>H<sub>2</sub>BzCO<sub>2</sub>H, is prepared from 3,5-dichlorophthalic anhydride, AlCl<sub>4</sub> and C<sub>4</sub>H<sub>4</sub>, free from thiophene, the mixture being heated finally at 90°; colorless, glassy lustrous prisms from C<sub>4</sub>H<sub>6</sub>, m. 168.5° (cor.). In conc. H<sub>2</sub>SO<sub>4</sub> the color is golden yellow. Yield, 95%. 1,4-Dichloroanthraquinone formula (I) below, is best prepared by adding the preceding acid to H<sub>2</sub>SO<sub>4</sub>

(monohydrate, 20 pts.), at 160°, the temp. is then reduced to 70-80° and ice added gradually, the temp. allowed to rize above 130°; orange-yellow needles from glacial AcOH, m. 187.5° (cor.). In conc. H<sub>4</sub>SO<sub>4</sub> the color is orange-yellow. Yield, 86.3%. 1,4-Diphenoxyanthraquinone, C<sub>20</sub>H<sub>16</sub>O<sub>4</sub>, is obtained from (I), dry PhOK and "natur" Cu, at the b. p.; yellow needles from alc., m. 165°. In conc. H.SO, the color is bluish violet. Yield, 93%. This compd. is identical with that of G. M. Walsh and C. Weizmann (C. A., 4, 2114) whose statements regarding its properties and method of prep. are incorrect. 1,4-Di-p-toluenesulfaminoanthraquinone, C14HaO2(NHSO4C7H7), is obtained from (I), p- olylsulfamide, K<sub>2</sub>CO<sub>2</sub>, Cu(OAc), and PhNO<sub>2</sub>; large, lustrous, reddish brown needles from glacia AcOH, m. 225°. Yield, nearly 200% of (I). In alc. NaOH the color is reddish violet. It is sol. in 200 pts. of b. glacial AcOH. 1,4-Diaminoanthraquinone, C<sub>14</sub>H<sub>2</sub>O<sub>2</sub>(NH<sub>2</sub>)<sub>2</sub>, is formed by gently warming the preceding compd. with H<sub>2</sub>SO<sub>4</sub> In glacial AcOH the color is bluish violet, in C<sub>2</sub>H<sub>4</sub>, or pyridine reddish violet. Yield, 92%. B. with PhNH2, AcOK and "natur" Cu converts (I) into 1,4-dianilinoanthraquinone, C14H4O2(NHPh)2; blue, lustrous plates with a brown surface tinge from glacial AcOH, m. 217°. The ordinary solns. are blue; in conc. H<sub>2</sub>SO<sub>4</sub> greenish blue. Yield, about 100% of (I). 1,4-Di-α-aminoanthraquinonylanthraquinone (II), is prepared in a similar manner to the preceding compd. from a-aminoanthraquinone, with PhNO, as solvent; violet needles with a metallic luster, not m. 410°. It is very sparingly sol.; the color in quinoline, or diphenylamine is violet, in conc. H<sub>2</sub>SO<sub>4</sub> dark green. Its vat-dye is reddish brown. Yield, 140% of (I). 4-Chloro-1-o-carboxyanilinoanthraquinone (III), is prepared by heating (I), during 15 hrs., at 155-60°, with anthranilic acid, anhydrous AcOK, Cu(OAc), and amyl alc.; bordeaux-red, interlaced needles, or plates, softens 258°, m. 262-3° (cor.). It gives a reddish violet color with pyridine + NH, and a dark green with conc. H,SO. Yield, 74.2%, which is increased to 92% if the heating is continued only 6-9 hrs., but in that case more than 50% of (I) is recovered unchanged. When treated with PCI and C.H. (free from thiophene) and then with AlCI, (III), gives 4-chloroanthraquinone-2,1-acridone (IV); lustrous, violet needles from pyridine, m. 267°. It dissolves in 10 pts. of pyridine with a red color, and in conc. H.SO, with a reddish brown shade. Yield, 78%. With aq. NaOH and Na hyposulfite (VI) gives a cherry-red vat dye, it colors cotton the same shade which, in air, changes to blue, then to reddish violet. 4-p-Toluenesulfaminoanthraquinone-2,1-acridone (V), is prepared by b. (IV) with p-tolylsulfamide, anhydrous AcOK, Cu(OAc), and PhNO, during 4 hrs.; small, bluish black needles from PhNO, m. 295°. It dissolves in 3000 pts. of b., glacial AcOH; in

pyridine, PhNH<sub>2</sub>, or PhNO<sub>2</sub> the color is intense blue. Yield, 80%. At 100°, conc. H<sub>2</sub>SO<sub>4</sub> hydrolyzes (V) to 4-aminoanthraquinone-2,1-acridone, C<sub>2</sub>H<sub>12</sub>O<sub>2</sub>N<sub>2</sub>; dark blue needles from PhNO<sub>3</sub>, not m. 410°. Yield, 84%. It dissolves in 15 pts. of b. PhNO<sub>2</sub> and gives a blue color with organic solvents. The vat dye is blood-red; cotton acquires from it the same shade, which changes to blue on exposure to air. 4-p-Toluidinoanthraquinone-2,1-acridone, C24H16O2N2, is prepared in a similar manner to (V) from (IV) and p-toluidine; bluish green, lustrous needles from PhNO2, m. about 300°. In organic media the color is green, in conc. H<sub>2</sub>SO<sub>4</sub> orange. Yield, 70%. The vat dye is violet; it colors cotton violet changing to bluish green in air. 4-a-Aminoanthraquinonylanthraquinone-2,1-acridone (VI), is prepared in a similar manner to (V) from a-aminoanthraquinone; violet, interlaced, very sparingly sol. needles, not m. 410°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is reddish brown. Yield, 75% The vat dye is blood-red; cotton acquires from it the same shade, which in air becomes violet, then bluish gray. 3,4-[or 5,6-]Dicklorobenzoylbenzoic acid, is prepared from 3,4-dichlorophthalic anhydride, in a similar manner to the isomer described above; small needles from C<sub>e</sub>H<sub>e</sub>, m. 216° (cor.). Yield, 96%. It is condensed by H<sub>2</sub>SO<sub>4</sub> (monohydrate) to 1,2-dichloroanthraquinone (VII); large, golden yellow needles from glacial AcOH, m. 208° (cor.).

In spite of its having the same m. p., it is not identical with Hammerschlag's compd. (Ber., 19, 1109 (1886)) which is not the 1,2-isomer. Yield, 95%. 4,5-Dichlorobenzoylbenzoic acid is obtained from 4,5-dichlorophthalic anhydride, AlCl<sub>2</sub> and C<sub>6</sub>H<sub>6</sub>; colorless, lustrous needles from C<sub>6</sub>H<sub>6</sub>, m. 209°. Yield, 98%. It is condensed by H<sub>2</sub>SO<sub>4</sub> (mono hydrate) to 2,3-dichloroanthraquinone (VIII), m. 267°. Yield, 92.3%. It is identical with Kircher's compd. from 1,2,3,4-tetrachloroanthraquinone (Ann., 238, 348 (1887)).

J. BISHOP TINGLE.

Action of Bromine and of Chlorine on Phenols: Substitution Products, Pseudobromides and Pseudochlorides. XXV. A Pseudobromide, Containing Sulfur, Derived from p-Cresol and its Transformations. Th. Zincke, W. Frohneberg and J. Kempp. Chem. Inst., Marburg. Ann., 381, 28-51; see C. A., 5, 1758.—2,5-Dibromo-3-thiomethyl-4-hydroxy(-oxy)toluene pseudobromide, formula (I), or (II), below, has been

described previously. It is best prepared from the perbromide (III), anhydrous AcOK

$$BrCH_{3}C \stackrel{CBr : C(SMe)}{CH \cdot CBr} COH$$

$$(I)$$

$$MeC \stackrel{CBr : C(SBr_{3}Me)}{CH \cdot CBr} COH$$

$$(III)$$

$$CBr : C(SBr_{3}Me) COH$$

$$(III)$$

and glacial AcOH. Yield, 53-60% of (III). With 1 N alkali hydroxide (I) gives a dark green Na salt. Pyridine converts (I) into a pyridinium bromide, the nitro derivative of which m. 205°. 2,5-Dibromo-3-thiomethyl-4-acetoxytoluene pseudobromide, BrCH<sub>2</sub>C<sub>2</sub>HBr<sub>2</sub>(SMe)OAc, from (I), Ac<sub>2</sub>O and conc. H<sub>2</sub>SO<sub>4</sub>; colorless, cubical crystals from MeOH, m. 136°. At the ordinary temp., moist acetone and (I) form 2,5-dibromothiomethyl-4-hydroxybenzyl alcohol, HOCH2C2HBr2(SMe)OH; lustrous plates from C<sub>2</sub>H<sub>2</sub>, m. 125-6°. It dissolves without decomp. in aq. Na<sub>2</sub>CO<sub>2</sub>, or NaOH; with HBr in glacial AcOH, it regenerates (I). 2,5-Dibromo-3-thiomethyl-4-hydroxybenzyl methyl ether, MeOCH<sub>2</sub>C<sub>2</sub>HBr<sub>3</sub>(SMe)OH, is prepared by b. (I) with MeOH; small, white needles from benzine, m. 81-2°. In cold, glacial AcOH, HNO, (d. 1.4) converts it into 2,5-dibromo-3-nitro-4-hydroxybenzyl methyl ether, MeOCH<sub>2</sub>C<sub>2</sub>HBr<sub>2</sub>(NO<sub>2</sub>)OH; long, yellow needles from benzine, m. 120-1°. Its alkali salts are red and sparingly sol. in H.O. The formation of this nitro compd. shows that (I) does not contain the group SBr:-CH<sub>2</sub>, SCH<sub>2</sub>Br, or SOMe. Glacial AcOH + AcONa convert (I) into 2,5-dibromo-3thiomethyl-4-hydroxybenzyl acetate, AcOCH2CeHBr2(SMe)OH; long, thick, colorless needles from MeOH, m. 137°. It does not give phenolic reactions and is, therefore, probably a quinone. It becomes green with alkalies and when b. with AcO, forms a diacetyl compound, C<sub>12</sub>H<sub>13</sub>O<sub>4</sub>SBr<sub>2</sub>, which is also produced from (I), Ac<sub>2</sub>O and AcONa; small, colorless needles from benzine, m. 131°. 2,5-Dibromo-3-nitro-4-hydroxytoluene pseudobromide, BrCH,C,HBr,(NO,)OH, from (I), glacial AcOH (4 pts.) and HNO, (d 1.4; 2 pts.), at the ordinary temp.; yellow needles from benzin, m. 118-9°. It becomes dark red with Na<sub>2</sub>CO<sub>2</sub>, or alkali hydroxide, and when b. with MeOH gives the 2,5-dibromo-3-nitro-4-hydroxybenzyl methyl ether described above. These reactions also show the structure of (I) (see above). 3,6,3',6'-Tetrabromo-5,5'-dithiomethylstilbene-4,4'-quinone (IV), is obtained by dissolving (I) in Et<sub>2</sub>O and shaking

$$OC \left\langle \begin{array}{c} CBr : CH \longrightarrow \\ C(SMe) : CBr \end{array} \right\rangle C : CHCH : C \left\langle \begin{array}{c} CH : CBr \longrightarrow \\ CBr : C(SMe) \end{array} \right\rangle CO$$

the soln. with AcONa, in H<sub>2</sub>O (1 N), or as anhydrous powder; deep black powder, m. about 240° (decomp.). It is practically insol. in the ordinary media. In presence of acetone it dissolves in aq., alkali hydroxides; with conc. H<sub>2</sub>SO<sub>4</sub> a dull green soln. is produced. Yield, about 40% of (I). Nitro derivative, from HNO, (d. 1.5) and (IV); in alkali its color is brown, changing to red. 4,4'-Diacetoxy-3,6,3',6'-tetrabromo-AcOC, HBr, (SMe)CH(OAc)CH(OAc)C, HBr, 5,5'-dithiomethyldiacetylhydrobenzoin, (SMe)OAc, is formed from (IV), Ac<sub>2</sub>O, glacial AcOH and conc. H<sub>2</sub>SO<sub>4</sub>; thick, colorless, lustrous crystals, m. 236°. MeOH alone, or in presence of conc. H<sub>2</sub>SO<sub>4</sub> does not react with (IV), but when 2 N aq. NaOH is added there is formed 3,6,3',6'-tetrabromo-5,5'dithiomethyl-4.4'-dihydroxydihydrobenzonyl dimethyl ether, HOC.HBr.(SMe)CH-(OMe)C2HBr3(SMe)OH; large, lustrous, colorless, rhombic crystals from glacial AcOH, m. 182°. Diacetyl derivative, C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>Br<sub>4</sub>S<sub>2</sub>, from Ac<sub>2</sub>O + conc. H<sub>2</sub>SO<sub>4</sub>; warty aggregates of small, white crystals from H<sub>2</sub>O, m. 220-2° (decomp.). In glacial AcOH,

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HCl and SnCl, reduce (IV) to 3,6,3',6'-tetrabromo-5,5'-dithiomethyl-4,4'-dihydroxy stilbene. HOC.HBr.(SMe)CH: CHC.HBr.(SMe)OH; small, colorless, lustrous plates from the Na salt in MeOH + AcOH, m. 245°. With Ac<sub>2</sub>O it gives a diacetyl derivative, C, H1,O, Br,S2; small, colorless needles from Ac,O, m. 280°. The dihydroxy:tilbene is converted, by HNO<sub>2</sub> (d. 1.4) and glacial AcOH, into (IV) (yield, 50%) together with a nitro derivative, which is separated by its greater sol. in glacial AcOH; small, yellow needles, m. 168°. In CHCI, HBr and (IV) form 3,6,3',6'-tetrabromo-5,5'-dithiomethyl-4,4'-dihydroxystilbene dibromide, NOC, HBr, (SMe)CHBrCHBrC, HBr, (SMe)OH; small, colorless needles from glacial AcOH containing a little HBr, m. 205° (decomp.). It easily regenerates (IV) by the action of MeOH, alc., moist acetone, or dil. AcOH. 3,6,3',6'-Tetrabromo-5,5'-dithiomethyl-4,4'-dihydroxydibenzyl, HOC.H-Br.(SMe)CH.CH.,C.,HBr.(SMe)OH, is formed together with (IV), from which it is separated by its greater sol. in Et.O; thin, lustrous plates and needles from glacial AcOH, m. 202°. It is sol. in alkali without decomp. Diacetyl derivative, CanH18O4Br4S, from AcO<sub>2</sub> + conc. H<sub>2</sub>SO<sub>4</sub>; small, colorless plates, or needles from C<sub>2</sub>H<sub>4</sub> or glacial AcOH, m. 219°. In glacial AcOH, HNO<sub>2</sub> (d. 1.4) converts the dibenzyl compd. into a dinitro derivative, C14HaOaN3Br4, no regeneration of (IV) occurs; small, yellow, lustrous crystals from tetrachloroethane, m. above 260° (decomp.). With Br, in tetrachloroethane, the dibenzyl compd. gives a perbromide, HOC.HBr. (SBr.Me)CH.CH.C.H-Br<sub>2</sub>(SBr<sub>2</sub>Me)OH; dark, orange-red needles from tetrachloroethane containing a little Br, decomp. about 150°, Br and HBr being evolved. When treated with NaHSO<sub>2</sub>, or b. with glacial AcOH, the perbromide regenerates the parent dibenzyl deriv. Aq. NaOH + CHCl, converts the perbromide into a black quinone, C18H10O2Br.Sp. which, although it resembles (IV) closely and gives the same reduction product, is regarded as being a stereoisomer of (IV). This second quinone is reduced more slowly than (IV). With HBr it gives a pseudobromide, which differs from that of (IV) chiefly in the fact that it regenerates the quinone with greater difficulty. J. BISHOP TINGLE.

Terpenes and Ethereal Oils. CV. Hydrogenation of Alicyclic Compounds. O. Wallach. Chem. Inst., Univ. Göttingen. Ann., 381, 51-95.—The reductions described below were carried out by means of Paal's method (C. A., 4, 314). Unless the material to be reduced is relatively pure, the Pd is likely to become inactive rather quickly. The method is by far the best hitherto employed for the reduction of terpenes; ethylene linkages are resolved irrespective of their relative position in the mol. and, with unsatd. ketones, the action may be regulated so that the CO is but little, if at all, affected and in any event no pinacolin is produced. p- $\partial$ -Menthanol, formula (XXXII) below, is obtained from d, d-d-terpineol (m. 35°); b. 209-10°; d<sub>20</sub> 0.905; n<sub>20</sub> 1.4629. Phenylurethan, C<sub>17</sub>H<sub>25</sub>O<sub>2</sub>N, crystals from MeOH, m. 117-8°. It is isomeric with Béhal's compd. (m. 94-5°) which is itself apparently a mixture of 2 forms. Active d-terpinol behaves like the inactive compd. d<sub>20</sub> 0.9000; d<sub>20</sub> 1.4619. Phenylurethan, m. 100-1°. When warmed with ZnCl<sub>2</sub> (I) is converted chiefly, or wholly, into  $\Delta$ -tetrahydro-d-cymene (carvomenthene) (II).

Carvomenthene gives a mixt. of nitrosochlorides, which are sepd. by acetone or MeOH; the more sparingly sol. m. 95-6°. With piperidine it forms a nitrolpiperidine; needles, from MeOH, m. 159°. In glacial AcOH, AcONa converts the nitrosochloride into d.l-carvotanacetoneoxime (III). This synthesis supplies a link to the chain of reactions by which it is possible to pass, without difficulty, from  $\beta$ -terpineol (IV) to tetra-

hydrocarvone (V). Sylveterpineol gives dihydrosylveterpineol (m-menthanol) (VI)

or (VII); b. 206-8°; d<sub>18</sub> 0-9090; n<sup>18</sup> 1.4645. Phenylurethan, crystals from dil. MeOH,

m. 71-4°. Pinol hydrate (VIII), is reduced to 2,8-menthandiol (IX), which should

be identical with the hydration product of dihydrocarveol (X). In order to avoid the production of resin it is best to hydrate (X) by means of highly dil. H<sub>2</sub>SO<sub>4</sub>. Both

the d- and l-modifications m.  $112-3^{\circ}$ ; b.  $265-70^{\circ}$ ; for the l-derivative, in alc.,  $[\alpha]_{0}^{16}$  — $21.07^{\circ}$ . A mixture of the d- and l-modifications m.  $108-9^{\circ}$ , whereas (IX), prepared by reduction, m.  $139-40^{\circ}$ . This result does not invalidate the formula for (IX), because the comp. contains 2 asymmetric C ats. and can, therefore, exist in several inactive modifications. Pineol is easily reduced to dihydropinol. Com. d-carvone is reduced, without difficulty, to l-tetrahydrocarvone. It was found that the d-earvone contained a little of the d,l-form, that d,l- and l-tetrahydrocarvoxime exert a marked mutual effect on each other's m. p. and that these oximes are separated with considerable difficulty. Eucarvone gives tetrahydroeucarvone, b.  $208-9^{\circ}$ ; d<sub>20</sub> 0.9065; \*\* $\frac{80}{1.4554}$ . The semicarbazone exists in 2 forms, the first m.  $201^{\circ}$  not  $191-2^{\circ}$  as stated previously; the second modification crystallizes with difficulty and m. indefinitely. a-Dicarvelone (XI), or (XII), is reduced in acetone with great ease, to tetra-

hydrodicarvelone, in which the H<sub>2</sub>C, : CMe groups of (XI), or (XII) become converted into Me<sub>2</sub>CH; long, flat crystals from alc., m. 129-30°. It is stable towards "perman-

ganate." Carvenolide,  $C_{10}H_{14}O_{2}$  (m.  $71-2^{\circ}$ ), absorbs, in MeOH, 1 mol. of H; the product is saturated, m.  $36-8^{\circ}$ ; b.  $260-1^{\circ}$ . It is probably a  $d_{i}l$ -form of pulegenic lactone (XIII). Contrary to the author's previous statement, which was based on its inability to combine with halogens, pulegenolide contains an ethylene linkage and, in MeOH, is reduced readily to dihydropulegenolide (XIV); long needles from MeOH, m.  $49-50^{\circ}$ ; b.  $260-1^{\circ}$ . It is volatil with steam and has a pronounced odor of men-

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thone. These results suggest that pulegenic acid is a mixt. of (XV) and (XVI), in

which the former predominates. It is (XV) which gives the hydroxylactone (XVII) (m. 129-30), from which pulenone is produced. Pulegenolid is formed from (XVII)

and it gives (XIII) when reduced, consequently it is regarded as being (XVIII), or (XIX). Pulegenolide, which gives (XIV), is probably (XX), or (XXI). Pulegenamide

is reduced to a compound, m. 149-50°. α-Fenchonitrile, C<sub>10</sub>H<sub>18</sub>N, cannot be freed completely from the  $\beta$ -compd. except by treatment with alc. KOH. When distilled, a portion decomp. The  $\alpha$ -nitrile, from d-fenchone, is d-rotatory; when reduced it gives dihydrofenchonitrile, C<sub>10</sub>H<sub>17</sub>N; liquid, b. 214-5°; d<sub>20</sub> 0.8730; n<sub>D</sub><sup>20</sup> 1.4434; a<sub>D</sub> --5°. The same compd. is obtained by distilling a mixt. of P<sub>4</sub>O<sub>10</sub> and dihydrofencholenamide. Mahla's "dihydrofenchonitrile" (Ber., 34, 3779 (1901)) is a mixt., the good agreement which he finds for the mol. ref. is due to an arithmetical error.  $\beta$ -Fencholenamide is reduced to fencholamide. Dihydro-β-fencholenonitrile, from the amide (m. 94°), has been obtained in a higher degree of purity than heretofore; b. 217-8°; d<sub>20</sub> 0.8680; n<sup>20</sup> 1.4426. α-Dihydrofencholenic acid, C<sub>10</sub>H<sub>12</sub>O<sub>2</sub>, is prepared from the amide and conc. HCl at 150° and is purified by means of its crystallin ammonium salt; b. 259-60°;  $d_{20}$  0.9740;  $n_D^{20}$  1.4583;  $\alpha_D$  2° 45′. Campholenonitrile is reduced to dihydrocampholenonitrile. Thuja ketone, Me,CHC(: CH,)CH,CH,Ac, is reduced, without a solvent, to dihydrothuja ketone, Me, CHCHMeCH, CH, Ac, which was purified by means of the semicarbazone; liquid, b. 185.5-6°; d<sub>20</sub> 0.8340; n<sub>D</sub> 1.4254; M 43.57. Semicarbazone, crystals from MeOH, m. 152-3°. In alc., the dihydro ketone is reduced to dihydrothuja ketol, Me<sub>2</sub>CHCHMeCH<sub>2</sub>CHMeOH; liquid, b. 191.5-2.5°; d<sub>18</sub> 0.835; n<sub>D</sub> 1.4352. With the MeMgI, the dihydro ketone gives the tertiary alcohol, Me,CHCHMeCH,CH,-CMe<sub>2</sub>OH; liquid with a pleasant odor of roses, b. 192-4°; d<sub>18</sub> 0.833; n<sup>20</sup> 1.4363. At

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155°, ZnCl<sub>2</sub> converts it into the hydrocarbon,  $C_{10}H_{20}$ ; liquid, b. 159.5-61°;  $d_{10}$  0.7575;  $m_D^{10}$  1.4315. Na  $\beta$ -thujaketonic acid, Me<sub>2</sub>CHC(: CHCO<sub>2</sub>Na)CH<sub>2</sub>CH<sub>2</sub>Ac, is reduced to  $\delta$ -acetyl- $\beta$ -isopropylvaleric acid (dihydrothujaketonic acid), Me<sub>2</sub>CHCH(CH<sub>2</sub>CO<sub>2</sub>H)-CH<sub>2</sub>CH<sub>2</sub>Ac; liquid. Semicarbazone, cryst. m. about 140°. The acid is oxidized by "hypobromite" to CHBr<sub>2</sub> and  $\beta$ -isopropyladipic acid, HO<sub>2</sub>CCH<sub>2</sub>CH<sub>2</sub>CHPr $^{\beta}$ CH<sub>2</sub>CO<sub>2</sub>H, which is purified with difficulty; crystals, m. 80-4°. Its calcium salt, when distilled, gives 1-isopropyl-3-cyclopentanone (XXII), which is also formed by the reduction of tanacetophorone (XXIII), by means of Pd. Semicarbazone, crystals from alc. m.,

190-1°. The tanacetophorone was prepared from Ca tanacetonedicarboxylate. Semicarbasone, crystallin, m. 187-8°. Isothujone (XXIV), is reduced, in MeOH, to thujamenthone (XXV). Thujone and α-thujaketonic acid, each of which contains a trimethylene ring, are not reduced in presence of Pd. Methylheptanone is easily reduced to methylheptanone (isoamylacetone), Me<sub>1</sub>CHCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>C; b. 164-5°; d<sub>10</sub> 0.8165; n<sub>10</sub><sup>10</sup> 1.4144. Semicarbazone, lustrous plates from MeOH, m. 157-8°. The saturated ketone exhibits but little tendency towards intramolecular condensation. The nitrosochloride (XXVI) (W. H. Perkin and O. Wallach, C. A., 4, 2446), is reduced

by means of Zn dust and AcOH to hexahydro-p-acetyltoluene (XXVII), a mixture of amines being formed simultaneously. A much better method of preparing (XXVII) consists in converting (XXVI) into  $\Delta^{4}$ -tetrahydro-p-acetyltoluene (XXVIII), which is very easily reduced by Pd to (XXVII).  $\Delta^{1}$ -Tetrahydro-p-acetyltoluene (XXIX), is not reduced by the ordinary methods, but with Pd and H it is converted quite easily into (XXVII). It has been shown previously that (XXIX) is prepared

without difficulty from  $\beta$ -terpineol (XXX), consequently this method in the best for the preparation of (XXVII). The properties of (XXVII) are as follows: b. 195-7°; d<sub>18</sub>

0.9055;  $n_0^{18}$  1.4509. Semicarbasone, m. 159°. A specimen which had remained during several mos. was then recryst. from MeOH, it was deposited in well-developed needles, m. 175°. Oxime, crystals from dil. MeOH, m. 57-9°;  $b_{14}$  125-30°. In alc., (XXVII) is reduced by Na to the alcohol (XXXI); liquid with a pleasant odor,  $b_{15}$  96°. Phenylurethan, crystals from MeOH, m. 62-3°. The best method of preparing hexahydro-p-toluylic acid consists in treating (XXVII) with "hypobromite" With MeMgI.

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(XXVII) gives p-menthane-3-ol (XXXII) (see above), which was purified by means of semicarbazine. Phenylurethan, crystals from MeOH, m. 95°. Another preparation, after repeated recryst. from alc., give 2 fractions, m. 116-7° and 89-91°, resp. They are probably stereoisomers. Acids cause colloidal Pd to flocculate and the presence of small quantities of impurity in the acid, or in the alkali, frequently render the Pd inactive. Consequently it is usually best to reduce amides in MeOH and not to employ the corresponding acids, or alkali salts. No special difference could be detected in the readiness with which the cyclohexeneacetic acids (XXXIII) and (XXXIV) were

reduced and the same is true of the 1,4-methylcyclohexeneacetic acids (XXXV) and (XXXVI). Both (XXXVI) and (XXXVI) yielded directly 1,4-methylcyclohexylacetic acid (XXXVII) in a high degree of purity; m. 73-4°, not 63-5°. It is practically cer-

tain that Paal's method of reduction will have a wide application and be of very great value for the conversion of unsatd. alicyclic acids into the corresponding satd. compds.

J. BISHOP TINGLE.

Terpenes and Ethereal Oils. CVI. Bicyclic Compounds from Cyclohexanone. O. Wallach. Chem. Inst., Univ. Göttingen. Ann., 381, 95-113.—The mol. ref. of cyclohexenehexanone (52.81) is not what would be expected if its formula were (I) below. In b. alc., Na reduces it to cyclohexenehexanol, C<sub>12</sub>N<sub>20</sub>O; m. 34-5°; b.

$$H_{3}C \underbrace{CH_{3} \cdot CH_{3}}_{CH_{3}} \cdot C: C \underbrace{CO \cdot CH_{3}}_{CH_{3}} \cdot CH_{3} \cdot CH_{3} \cdot CH_{3} \cdot CH_{4} \cdot CH_{3} \cdot CH_{4} \cdot CH_{5} \cdot CH_{5$$

272-3°;  $d_{s7}$  0-974;  $n_{B}^{37}$  1.5807;  $d_{19}$  0.989;  $n_{B}^{19}$  1.5071. It is also formed from Na and cyclohexanone, in toluene, at 100°. *Phenylurethan*, crystals from MeOH, m. 118-9°. With CrO<sub>2</sub> + H<sub>2</sub>SO<sub>4</sub> the alc. regenerates (I), but in C<sub>2</sub>H<sub>2</sub> at 0°, KMnO<sub>4</sub> (2%) oxidizes it to a trihydroxy alcohol,  $C_{12}H_{19}(OH)_3$ ; cryst.  $b_{19}$  202-5°. It could not be purified completely. A ketonic acid (III) is formed, together with the preceding alc.; it is the

chief product of the reaction and is extracted from the mixt. of K salts by means of Et<sub>2</sub>O + H<sub>2</sub>SO<sub>4</sub>. Yellow needles from C<sub>6</sub>H<sub>4</sub>, m. 74-5°; b<sub>14</sub> 205-15°. It may be sublimed. Silver salt. Semicarbasone, crystals from MeOH, m. 190-200°. Chlorohydrate, C<sub>12</sub>H<sub>19</sub>O<sub>2</sub>Cl, from (III) and dry HCl, in glacial AcOH, at the ordinary temp.; white plates from ligroin, m. 48-51°. It reacts with difficulty with "hypobromite" and herefore does not contain Ac. Hitherto it has not been possible to obtain (III) directly from (I) and "permanganate." Cyclohexenehexanoneoxime, m. 146-51°. It is reduced to bicyclohexenehexylamine, C<sub>12</sub>H<sub>11</sub>N; solid, m. 33-5°. It volatilizes only slowly with steam. Hydrochloride, C<sub>11</sub>H<sub>12</sub>N.HCl, from dry HCl, in Et<sub>2</sub>O; white crystals. "Permanganate" oxidizes the amine to (III). In view of these facts it is impossible to decide between (I) and (II) as the formula for cyclohexenehexanone, but, on the whole, the evidence appears to favor (I), otherwise we are led to the assumption

of the existence of a secondary alc. which eliminates H<sub>2</sub>O very readily, although it cannot be oxidized to a ketone. In MeOH, (I) is reduced easily by Pd and H to cyclohexylhexanone (IV) (see preceding abstr.); liquid, b<sub>12</sub> 137°; d<sub>18</sub> 0.978; n<sup>16</sup> 1.4887.

Semicarbasone, m. 203°. Oxime, m. 100°. Benzylidene derivative, C<sub>10</sub>H<sub>M</sub>O, lustrous plates from alc., m. 100°. Na reduces (IV) to cyclohexylhexanol (V), which is also formed from Pd, H and cyclohexenehexanol (see above); solid, b. 264°; m. 42°. Phenylwrethan, m. 117°. At 190°, ZnCl<sub>2</sub> converts (V) into cyclohexylhexene (VI); liquid, b.

237°;  $d_{50}$  0.9010;  $w_{30}^{20}$  1.4910; mol. ref. 52.72. Yield, good. Nitrosochloride, crystals from alc., m. 140°. In glacial AcOH, CrO<sub>3</sub> oxidizes (IV) to an acid (VII), which is also formed from (III), Pd and H, in dil. alkaline soln.; crystals from ligroin + Et<sub>3</sub>O, m. 58°. It may be distilled under reduced pressure. Semicarbazone, needles from MeOH, m. 172-3°. It is utilized for the purification of (VII). Oxime, slender

needles, m. 105°. Methyl ester, from the acid, MeOH and dry HCl, at the ordinary temp.; liquid, b<sub>18</sub> 173°; d<sub>18</sub> 1.0175; n<sup>18</sup> 1.4659. Yield, almost quant. No ketone could be obtained from (VII) and soda-lime; it is not oxidized by CrO<sub>2</sub>, nor does it react with HCl, at 190°, nor with dil. H<sub>2</sub>SO<sub>4</sub>, at 160°. In dry toluene, Na and the Me ester, at 120-30°, give 1-hexahydrobenzoyl-2-cyclopentanone probably (VIII), which was purified by means of its Cu salt; colorless oil, b,, 150°. It dissolves in aq. NaOH (10%) and gives a violet color with FeCl, in dil. alc. Yield, 50% of (VII). Copper salt, grayish green and pulverulent. No semicarbazone or oxime could be obtained from (VIII); with b. aq. NaOH it regenerates (VII). Methyl derivative, probably (IX) is obtained by the interaction at 100°, of MeI and the Na compd., which is prepared from (VIII), Na and toluene, at 130°; liquid, b<sub>18</sub> 150°. It does not dissove in aq. NaOH, gives no color with FeCl<sub>2</sub> and does not form a Cu salt. Semicarbazone, C<sub>12</sub>H<sub>22</sub>-O<sub>2</sub>N<sub>2</sub>, crystals from MeOH, m. 203°. In alc., Na reduces (VII) to a hydroxy acid, C12H202; viscid liquid. When distilled it passes into the lactone, C12H202; crystals, m. 45°; b<sub>12</sub> about 175°. With H<sub>2</sub>SO<sub>4</sub> (1:1.5) this lactone gives an unsaturated acid, C12H20O2; liquid, b20 182-6°. Silver salt. At 230°, aq. KOH converts (VII) into a mixture of acids, the chief constituent being an unsaturated acid which volatilizes with difficulty, m. 172°. In moist Et<sub>2</sub>O, Na reduces cyclohexanone to a mixture of cyclohexanol and cyclohexanonepinacol (cyclohexanonepinacone; 1,1'-bicyclohexyldiol) (X). After b. during 2 hrs. with dil. H<sub>2</sub>SO<sub>4</sub>, it is converted into  $\Delta^{1,1'}$ -bi-

$$H_1C$$
 $CH_2 \cdot CH_3 \cdot CH_3 \cdot C(OH)C(OH)$ 
 $CH_2 \cdot CH_3 \cdot C$ 

eyclohexene (XI); liquid, b.  $250-3^{\circ}$ ;  $b_{15}$   $110-5^{\circ}$ ;  $d_{20}$  0.9485;  $n_{20}^{20}$  1.5287; mol. ref. 52.65; this is greater by 0.31 than the calculated value, the difference being due to the proximity of the 2 ethylene linkages. In glacial AcOH, (XI) and HBr form a dibromohydrate,  $C_{12}H_{20}B_{12}$ ; white needles from MeOH, m.  $68-9^{\circ}$ . Cyclohexanol,  $C_{2}H_{11}OH$ , forms a

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stable compound with CaCl<sub>2</sub>. Its production may be utilized to purify the alc. and also to separate any of it which may be present in com. cyclohexanone. 1,3-Dimethyl-

dicyclohexene is prepared in a similar manner to (XI), from 1,3-methylcyclohexanone; liquid, b. 265-7°.

J. BISHOP TINGLE.

Phenol-m-sulfonic Acid, its Isolation and its Nonformation from Phenol and Sulfuric Acid. JULIUS OBERMILLER. Inst. Electrochem, and Techn. Chem., Roy. Techn. Ann., 381, 114-22.—Phenol-m-sulfonic acid is best prepared. Hochsch., Stuttgart. in a condition of purity, by diazotizing m-sulfanilic acid, the resulting soln., after neutralizing with PbCO<sub>3</sub>, gives the Na salt, which is then converted into the magnesium sall, (HOC2H4SO2)2Mg.6H2O; long, very thin, brittle, pale yellow leaflets. When treated with Br it does not eliminate H.SO.. By means of this salt it is possible to detect the presence of even traces of the m-sulfonic acid in mixtures of the isomers. Sodium sall, aggregates of broad, brittle plates, m. 300-10°. Zinc sall, thin, flat, pointed prisms. Lead sall, does not deposit PbSO4 when its aq. soln. is allowed to remain at the ordinary temp. The barium, strontium, calcium, copper, aluminium and potassium salts have no great degree of crystallizing power. Of the salts containing the complex -OM"SO<sub>2</sub>- (M = metal), from the OH and SO<sub>2</sub>H groups, the barium, strontium, calcium, potassium and sodium salts are fairly stable, but crystallize with great difficulty; the magnesium, zinc, copper and ammonium salts decomp., especially when the temp. is raised. The mono (SO<sub>2</sub>) salts do not liberate PhOH when heated. Phenol-m-sulfonic acid, in neutral soln., gives a violet color with FeCl, which is discharged by warming, or by adding acid. The color has about the same intensity as that given by the p-acid; it is much more intense than the tint obtained from PhOH, but greatly inferior to the color produced by the o-acid. Basic lead salt is best prepared from the Na salt, Pb(OAc), and Pb(OH), white and sparingly sol. Dil. H<sub>2</sub>SO<sub>4</sub> dissolves it, the soln. containing the mono-Pb salt. By the interaction of PhOH and H<sub>2</sub>SO<sub>4</sub> (100%) (1.5 pts.), at 15-20°, during 6 hrs., there are produced chiefly the o-(40%) and the p-sulfonic acids (60%). With 1.15 pts. of the H<sub>2</sub>SO<sub>4</sub> at 90-100°, during 6, 20, or 100 hrs., the product consists of the p-sulfonic acid. In all cases a little 2,4-disulfonic acid was formed and some PhOH escaped sulfonation. Not the slightest trace of m-sulfonic acid could be detected in the products from any of the expts. Details are given of a method for sepg. the mixed sulfonation products; by its means very small quantities of the m-acid can be detected, through the production of the Mg salt described above. J. BISHOP TINGLE.

Walden's Inversion and Substitution Processes. EMIL FISCHER. Chem. Inst., Univ. Berlin. Ann., 381, 123-41.—A number of examples of Walden's inversion are given, they may be summarized as follows: NOBr converts d-alanine into l-bromopropionic acid, but with d-alanine ester it gives d-bromopropionic ester. Similar differences in behavior are shown in the case of leucine, phenylalanine and aspartic acid and their respective esters. Ag<sub>3</sub>O converts l-bromopropionic acid into l-lactic acid, but with l-bromopropionylglycine, followed by hydrolysis, it gives d-lactic acid. d-Bromoisovaleric and NH<sub>3</sub>, give l-valine, but d-bromoisovalerylglycine and NH<sub>3</sub>, followed by hydrolysis produced d-valine. In the majority of cases, however,  $\alpha$ -halogen acids of the aliphatic series, their esters and their condensation products with glycocoll give, by treatment with NH<sub>3</sub>, products which are optically identical. Similar differences have been observed in the action of PCl<sub>5</sub>, in that of PCl<sub>5</sub> as compared with PBr<sub>5</sub> and in the removal of halogens, different results being obtained by the use of Ag<sub>2</sub>O, or of

aq. KOH. Examples are also cited of similar results in the case of aliphatic acids substituted in the  $\beta$ -position, altho the expt. work in this series is much smaller in quantity. Hitherto the investigations have been confined to acids or their immediate derivs. In spite if these gaps in the expt. record, but subject to them, the author concludes that the Walden inversion is a general phenomenon and that it is related, in the most intimate manner, to the mechanism of substitution. This latter is regarded as being preceded by the formation of additive compds. (Kekulé's "polymols."), in consequence of the activity of subsiduary valencies. The entering group, or at., need not necessarily take place, on the C at., of the group or at. which it substitutes, it is at least equally probable that it will occupy some other position. Obviously, its eventual location can only be detd. in the case of substances containing an asymmetric C at. In other words, the Walden inversion is not to be considered as a rearrangement, in the ordinary sense of the term, but as being an entirely normal process which, in general, occurs, or does not occur, with equal readiness. The question as to whether a given substitution will, or will not, be accompanied by a change in the configuration of the mol, depends on the nature of the reaction employed and on that of the other groups which are linked to the asymmetric C at. Optical antipodes possess an equal energy content and therefore their stability is identical, consequently, so far as the energy factor is concerned, there is an equal probability of either optical isomer being formed in the course of a given reaction. Therefore a substitution process, applied to an optically active compd., may be accompanied by (a) complete inversion, (b) partial inversion, (c) no inversion. In the case of (a) and (c) the substitution product will be optically active, whereas if (b) applies a complete, or partial d<sub>i</sub>l-derivative will be obtained. The changes indicated above cannot be followed conveniently by the use of the usual at. models of the Kekulé-v. Baeyer type, because they are too static. Illustrations are given of a new form. The substituents are represented by celluloid balls, attached to pieces of cork, which carry bristles. For the C ats. are employed small, wooden spheres, also covered with bristles; by this means the balls may be attached, removed, or caused to migrate with ease and, by means of other bristles, provision is made for the representation of the subsidiary valencies. The facts collected during the study of the Walden inversion explain the failure of I. Wislicenus to det. the configuration of the compds. resulting from the addition of halogens, or of halogen acids to unsaturated compds. That similar contradictions have not been encountered among the members of the sugar group, including the acids and alcs., is due to the author's instinctive avoidance of any reaction which would cause substitution in the asymmetric C at. Conclusions, similar to those given above, have been reached independently and by a study of entirely different expt. material, by A. Werner (C. A., 5, 2453). The paper concludes with a criticism of Nef's methylene theory. Optically active a-bromopropionic acid and NH, could only give d,l-alanine if Nef's theory were correct, because it postulates the intermediate production of the complex > CMeCO<sub>2</sub>H, which is, of course, not asymmetric. As is well known, optically active alanine is obtained by the reaction in question. In order to prop up the methylene theory, Nef has stated in a review (J. Am. Chem. Soc., 30, 645 (1908)) that the valencies of the C at. are dissimilar, 2 being - and 2 +. This would lead, in the case of the reaction mentioned above, to the formation of the complexes (I) and (II) below,

from which, by the addition of NH<sub>2</sub>, 2 optically active alanines would be formed. This hypothesis of Nef is without experimental evidence; it would involve the assumption of the existence of 2 stereoisomeric propionic acids.

J. BISHOP TINGLE.

Relationship between Perbromides and Bromo Substitution Compounds, Observed in the Case of Acetylacetonecarbamide and its Tautomers. O. STARK. Chem. Lab., Univ. Kiel. Ann., 381, 143-99.—In order to avoid confusion, it may be well to state that the author uses the term "perbromide" as a class name and distinguishes between "dibromo-" and "tetrabromoperbromides" containing 2 and 4 ats. of Br, resp. Acetylacetonecarbamide dibromoperbromide, formula (I) below, is prepared from yellow acetylacetonecarbamide (II) and Br, in CHCl<sub>2</sub>, or distilled, aq. HBr; slender, lustrous,

$$OC \left\langle \begin{matrix} NBr_2 = CMe \\ | \\ N: CMe. CH_2 \end{matrix} \right\rangle OC \left\langle \begin{matrix} N: CMe \\ N: CMe \end{matrix} \right\rangle CH_2 OC \left\langle \begin{matrix} NH \cdot CMe(OH) \\ NH \cdot CMe(OH) \end{matrix} \right\rangle CBr_2$$
(II) (III)

orange-yellow needles, becomes black 300°, not m. 350°. Yield, about 110 and 217% of (II), resp. At the ordinary temp., a small quantity of H<sub>2</sub>O converts (I) into 4,6-dimethyl-4,6-dihydroxy-5-dibromo-2-ketohydropyrimidine (III) and 4,6-dimethyl-5-bromo-2-ketopyrimidine (IV), which was separated as the Na salt. With a large excess of H<sub>2</sub>O, (I) gives (IV) only. 4,6-Dimethyl-2-ketopyrimidine (V) is formed

$$OC \left\langle \begin{array}{c} N : CMe \\ N : CMe \\ \hline \\ (IV) \end{array} \right\rangle CHBr \qquad OC \left\langle \begin{array}{c} N : CMe \\ NH \cdot CMe \\ \hline \\ (V) \end{array} \right\rangle CH \qquad OC \left\langle \begin{array}{c} NBr_2 = CMe \\ NH \cdot CMe = \\ \hline \\ (VI) \end{array} \right\rangle CH$$

by passing CO<sub>2</sub> into a soln. of (II) in abs. alc. EtONa; colorless, lustrous needles from acetone, m. 200°. Yield, 62.5% of (II). In dry CHCl<sub>2</sub>, Br converts it into 4,6-dimethyl-2-ketopyrimidinediperbromide (VI); becomes gray 160°, chars without m. 360°. Yield, 212% of (V). With H<sub>2</sub>O it behaves as does (I), giving with a little H<sub>2</sub>O, (IV) and with excess, (III) and (IV). Under the conditions described above, in CHCl<sub>2</sub>, or aq. HBr, with Br in excess, (II) gives acetylacetonecarbamide tetrabromoperbromide (VII); microscopic needles, chars without m. 220°, darkening previously. It is con-

$$OC \begin{picture}(100,0) \put(0,0){\line(1,0){100}} \put(0,0){\line(1,0)$$

verted into (III) and (I) by H<sub>2</sub>O. The "bromohydroxy compd." from (II), and Br, in AcOH, and the "bromoethoxy compd." from (I) and abs. alc., described previously (C. A., 3, 1174) are found to consist essentially of acetylacetonecarbamide hybrobromide, which is prepared directly from (II) and conc., aq. HBr, in presence of glacial AcOH; while needles from alc., becomes colored 240-50°, softens about 330°, decomp. and evolves gas 345°. Sodium 4,6-dimethyl-5-bromo-2-ketopyrimidine (VIII), is obtained from (II) and aq. NaOBr; slender, white needles from H<sub>2</sub>O, becomes colored 325°, decomp. violently 336-8°. Yield, 166% of (II). B., dil. AcOH converts (VIII) into 4,6-dimethyl-5-bromo-2-ketopyrimidine (IX); long, yellow prisms from dil. AcOH, darkens 220-2°, decomp. 228-31°. In addition to this form (IX) exists also in a white modification (X), which is obtained by warming (VIII) with H<sub>2</sub>O and adding dil. AcOH

gradually; lustrous, white prisms from H<sub>2</sub>O. When heated it behaves like the yellow form. When (IX) is dissolved in H<sub>2</sub>O and a few drops of aq. NH<sub>2</sub> added (X) crystallizes out. The reverse change, white (X) to yellow (IX), is caused by prolonged heating with dil. AcOH. Nitrate (XI), from (X), in abs. alc., and conc. HNO<sub>2</sub>, at a low temp., colorless, lustrous needles, becomes colored 160°, chars without m. 200°.

Yield, quant. Dissitrate (XII), is prepared by dissolving (IX), or (X), in 2 N HNO<sub>2</sub>;

$$OC$$
 $N(HNO_s): CMe$ 
 $CHBr$ 
 $OC$ 
 $N(HO_s): CMe$ 
 $N: CMe$ 

reddish yellow crystals with 2 H<sub>2</sub>O, stable in absence of air, darkens 160°, explodes vigorously 196-7°. Bromacttylacetonecarbamide dibromoperbromide (XIII), is prepared from either (IX) or (X) and Br, in CHCl<sub>2</sub>, or aq. HBr; small crystals, darkens 160°,

chars without m. 200°. Yield, 160% of (IX) or (X). With H<sub>2</sub>O it gives (III). 4,6-Dimethyl-5-bromo-2-hetopyrimidine dibromoperbromide (XIV), is prepared from Br and (X), in CHCl<sub>2</sub>; yellow and unstable. It is converted into (III) by H<sub>2</sub>O. At the ordinary temp. (XIV) transforms spontaneously into 4,6-dimethyl-5-dibromo-6-bromo-2-hetopyrimidine (XV), which is prepared more conveniently from (X), Br and CHCl<sub>2</sub>, at 30-40°, until HBr just begins to be evolved; yellow needles, darkens above 220-40°, chars without m. 300°. Yield, 145% of (X). Apparently the comp. was not purified completely. At the ordinary temp., H<sub>2</sub>O converts (XIV) into a mixt. of (III)

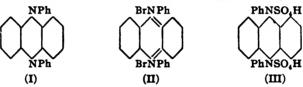
and 4,6-dimethyl-5-dibromoacetylacetonecarbamide (XVI), which is sepd. by its solubility in CHCla; slender, white, microscopic needles, darkens 140-50°, blackens 160°, decomp. 160-70°. Yield, 14.4% of (XIV). The aq. mother liquor obtained during the decomp. of (XIV) contains bromoacetylacetonecarbamide hydrobromide, C.H.ON, Br.-HBr, which was also prepared for comparison directly from the bromocarbamide; bundles of needles from MeOH; darkens 180°, chars without m. 300°. Yield, 33% of (XIV). Bromoacetylacetonecarbamide tetrabromoperbromide (XVII), is formed from Br and (IX), in aq. HBr; aggregates of red needles, darkens 160°, chars without m. 200°. It is converted by H<sub>2</sub>O into (III), the reaction being quant. The preparation of (III) from (II) and Br, in H<sub>2</sub>O, has been described previously; (III) cannot be cryst. from alc. or MeOH, because, contrary to the statement of Evans (J. prakt. Chem., 48, 494 (1893)) a portion decomp. It becomes gray 110-40° and chars without m. 160°. Prolonged heating, at 100°, also causes it to blacken. At the ordinary temp., NaOH (30 g.) and H<sub>2</sub>O (800 cc.) decomp. (III) (50 g.) into HBr, CO<sub>2</sub>, NH<sub>2</sub>, AcOH and lactic acid. With 2 mol. of NaOH, (III) forms a sol. sodium salt. At the b. p., Br in CHCla. converts (II) into a mixture of pentabromo-4,6-dimethyl-2-ketohydropyrimidine (XVIII), with the heptabromide (XI) (see below) and (III). The heptabro-

mide is extracted by means of C<sub>0</sub>H<sub>4</sub>, and (XVIII) by the help of CHCl<sub>2</sub>. From C<sub>0</sub>H<sub>4</sub>, or CHCl<sub>3</sub> (XIII) is deposited in pale yellow, well developed prisms, m. 183-4° (decomp.). It is stable towards H<sub>2</sub>O and with aq. or alc. 2 N NaOH gives a sodium salt; crystallin. In dry CHCl<sub>3</sub>, (XVIII) and dry HBr form an additive compound; yellow, crystalline and unstable. Pentabromo-4,6-dimethyl-2-ketohydropyrimidine dibromoperbromide (XIX), is formed together with (XVIII), as above, or from it by the

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action of Br, in CHCl<sub>2</sub>; yellow, interlaced needles. Yield, 150% of (XVIII). It is rather unstable and is converted completely into (XVIII) by the action of abs. alc., or of dil. AcOH, at the ordinary temp. The same change, which is, however, incomplete, takes place by  $\mathfrak{ge}$ cryst $\mathfrak{o}$ . (XIX) from  $C_0H_0$ . When heated with CHCl<sub>2</sub> and Br, in excess, (II), (V), (IX) and (X) give, in each case, (XVIII).

Aromatic Hydrazines. IX. Tetraphenylhydrazine and Hexaphenylethane. HEINRICH WIELAND. Chem. Lab., Acad. Sci., Munich. Ann., 381, 200-16.—When b. with toluene (5 pts.), during 0.5 hr., in an atmp. of CO, tetraphenylhydrazine is converted into a mixture of Ph<sub>2</sub>NH, diphenyldihydrophenazine, formula (I) below and o-anilinotriphenylamine. These are separated as follows: the toluene is distilled and the residue treated with Et<sub>2</sub>O, from this solution warm gasoline ppts. the phenazine and tert. amine. The oily ppt. is treated repeatedly with gasoline, then dissolved in Et<sub>2</sub>O and (I) pptd. by the addition of alc. The phenazine is purified by means



of  $C_8H_8$  + alc.; very slender, colorless needles, m. 172-5°; mol. wt., 919. The compd. soon turns green in air, because of its conversion into an o-quinoidal salt. Yield, 50% of the hydrazine. Quinoidal dibromo derivative (II), from (I) and Br, in C<sub>6</sub>H<sub>6</sub>; dark green, granular, amorphous powder. With Zn dust, glacial AcOH and alc. it regenerates (I). o-Anilinotriphenylamine, Ph.NC.H.,NHPh (see above), is purified thru the hydrochloride; amorphous, m. 85°. Hydrochloride, from dry HCl, in Et<sub>2</sub>O; crystallin. The constitution of the amine is shown by the fact that Br converts it into (II) and HBr. The yield of Ph<sub>2</sub>NH, as above, is 45% of the hydrazine. These results explain the reactions which occur in the formation of the well known blue color, obtained by treating Ph,NH, in conc. H,SO,, with an oxidizing agent, especially HNO, or HNO. The color is given much more readily by (I) than by Ph,NH, in conc. H<sub>2</sub>SO<sub>4</sub>. The color is due to o-quinoidal phenoperazonium acid sulfate (III). When dil. carefully with H,O the normal, green sulfate is deposited. The acid converts the Ph.NH into tetraphenylhydrazine, which decomp. into Ph.NH and diphenylhydroxylamine, these then condense to (I) and pass into (III). At 90-5°, in toluene, NO, in absence of air, converts tetraphenyl- or tetra-p-tolylhydrazine into the resp. diarylnitrosoamines, R2NNO, which were fully identified. At about 130° they eliminate NO almost quant., and give diarylamine (60-90%), together with a phenazine deriv. At 90-5°, in toluene, tetraphenylhydrazine and Ph<sub>2</sub>C combine, in an atm. free from air, to form triphenylmethyldiphenylamine, Ph.CNPh; large, lustrous needles from toluene, m. 172°. Yield, 74%. Conc. H<sub>2</sub>SO<sub>4</sub>, which gives a greenish yellow color with the amine, decomp. it into triphenylcarbinol and PhNH. In b. xylene the amine is resolved into Ph.C and tetraphenylhydrazine. Tetra-p-tolylhydrazine reacts in a similar manner with Ph.C, giving triphenylmethyl-p-ditolylamine, Ph.CN-(C<sub>8</sub>H<sub>4</sub>Me)<sub>2</sub>; colorless crystals from C<sub>6</sub>H<sub>6</sub> + alc., m. 164°. Under the influence of the cathode rays, in a vacuum, at the temp. of liquid air, tetraphenyl-, sym. p-ditolyl-, p-dianisyldiphenyl- and tetra-p-biphenylhydrazines exhibit a blue phosphorescence, and the first three compounds soon acquire an intense green color, which quickly disappears, when the rays are cut off. Tetra-p-biphenyl- and tetra-p-tolylhydrazines, under the conditions mentioned, acquire a temporary, dark yellow color. In other respects the compds. are not changed. The color of diphenylnitrosoamine is not perceptibly affected by the rays, but benzophenoneketazine acquires a faint, permanent,

gray shade, because of slight decomp. The reactions described above are explained on the assumption that the tetraarylhydrazines are dissociated, primarily, into the complex R<sub>2</sub>N, exactly as hexaphenylethane is resolved into triphenylmethyl, the subsequent changes, in both cases, being similar in their nature (see following abstr.).

J. BISHOP TINGLE.

Aromatic Hydrazines. X. Tetrabiphenylhydrazine. HEINRICH WIELAND AND ARTHUR SUSSER Chem. Lab., Acad. Sci., Munich. Ann., 381, 217-29; see preceding abstr.—Di-p-biphenylamine, (PhC,H,),NH, is prepared by b., during 24 hrs., a mixture of p-iodobiphenyl, p-acetylaminobiphenyl, K<sub>2</sub>CO<sub>3</sub>, Cu bronze and PhNO<sub>4</sub>, the resulting Ac compd. being then hydrolyzed by means of b., alc. KOH; colorless, pearly lustrous plates from C<sub>e</sub>H<sub>e</sub> or xylene, m. 209°. Yield, about 90% of the I compd. Hydrochloride, long prisms from Et.O + dry HCl. Acetyl derivative, cryst. The soln of the amine in conc. H<sub>2</sub>SO<sub>4</sub> is colorless, with a nitrite it becomes brownish red, changing to green. Nitrosamine, C14H18ON2, from the amine and NaNO2, in glacial AcOH; yellow needles from CoH, decomp. 172°. Yield, nearly quant. At 10°, finely divided KMnO, oxidizes the amine, in acetone, to tetra-p-biphenylhydrazine, (PhC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>NN(C<sub>6</sub>H<sub>4</sub>Ph)<sub>2</sub>; colorless, cryst. crusts from C<sub>6</sub>H<sub>6</sub> + abs. alc., m. 165°. Yield, 80%. It gives a characteristic, intensely violet color when warmed with glacial AcOH. In xylene the color is yellow, changing to brown. In b. toluene it decomp, less readily than tetraphenyl- or tetra-p-tolylhydrazines. At 10°, ethereal HCl and the hydrazine give a green, unstable dihydrochloride, which the author formulates as a quinoneimmonium chloride, formula (I) below, because, with dil. alkalies, it regenerates the

$$PhCH \xrightarrow{CH : CH} C : NCl(C_{\bullet}H_{\bullet}Ph)NCl(C_{\bullet}H_{\bullet}Ph) : C \xrightarrow{CH : CH} CHPh$$

$$(I)$$

hydrazine immediately and it is formed more slowly at 0° than at 10°. At the ordinary temp., the dihydrochloride changes into a mixture, from which the following compds. were isolated. *Dichlorobiphenoperasine* (II), is extracted from the crude solid

by b. alc. containing a little NH<sub>2</sub>, or alc. KOH; yellow needles from xylene, not changed 380°. The nature of this compd. is proved by the fact that when b. during 8 hrs., with Na and amyl alc., it is reduced to biphenoperazine, which is also present after long standing of the mother liquor which is obtained during the oxidation of dibiphenylamine. Dihydrochloride (III), from (II) and dry HCl, in Et,O; violet crystals with a bronze luster. o-Biphenylaminotribiphenylamine, (PhC,H,)2NC,H,PhNHC,H,Ph, is formed together with (II) and is sepd. with difficulty, from the di-p-biphenylamine, by its smaller sol. in CaHa; colorless, interlaced needles from xylene, m. 275°; it gives an intense green color with FeCl, or Br. o-Chlorodibiphenylamine, PhC,H,ClNHC,H,Ph, is formed together with the preceding compd. and is the most readily sol. portion of the mixture; heavy, opaque, poorly developed crystals from alc., m. 119° to a turbid liquid, which becomes clear 130°. With Br, in CHCl, the tetra-p-biphenylhydrazine gives an unstable, dark green bromide. This soon changes to a mixture of di-p-biphenylamine and a dibromodi-p-biphenylamine, C24H17NBr2, which are separated by shaking with H<sub>2</sub>O, evaporating the aq. sol. and extracting the residue with C<sub>6</sub>H<sub>8</sub>; long, lustrous needles from C<sub>6</sub>H<sub>6</sub> + alc., m. 151°. These results show that tetra-p-

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biphenylhydrazine resembles the p-tolyl rather than the Ph compd. It is to be understood that the biphenyl groups  $PhC_0H_4$  are all -p-. The representation of the above bidiphenylaminotribiphenylamine and chlorodibiphenylamine, as o-compds., appears to be essentially hypothetical.

J. BISHOP TINGLE.

Acid Hydrolysis of Starch (DURYEA). 28.

Alcoholic Fermentation (LEBEDEV). 11.

Capillary Rise of Amines, etc. (SKRAUP, PHILIPPI). 2.

Carbon Ditelluride (STOCK, BLUMENTHAL). 6.

Conductivity of Organic Acids (WIGHTMAN, JONES). 2.

Cotton (MOSENTHAL). 24.

Determination of Amino Groups (VAN SLYKE). 11.

Mechanism of Saponification (MEYER). 27.

Photokinetics of Bromine Substitution (BRUNER). 2.

Physical Chemistry Measurements (CASTANARES). 2.

Reactions of Carbon Dioxide (MEYER, SCHUSTER). 6.

Yeast Gum (EULER, FODER). 11.

Unna, E.: Synthese einiger Galactoside. Berlin: E. Ebering. 8°, 40 pp., 0.75 M.

## 11. BIOLOGICAL CHEMISTRY.

W. J. GIBS.

Some Thoughts on the Action of Enzymes with Special Reference to the Nature of Pepsin. J. E. Hancock. Am. J. Pharm., 83, 373-6.

H. C. FULLER.

The Forensic Testing of Veronal. A. HEIDUSCHKA. Arch. Pharm., 249, 322.—
In a case of veronal poisoning the amt. of the substance found in the body was small and was scattered thru the different organs. The greater part taken into the system had been eliminated before death.

H. C. Fuller.

The Influence of Guaiacol Derivatives on the Secretion of Glucuronic Acid. T. KNAPP. Schweiz. Wochschr., 49, 229-31, 245-8, 257-61.—The administration of guaiacol and its derivs. increases the secretion of glucuronic and ethylsulfuric acids; the increase varies with the compd. ingested and is apparent over different intervals. In the case of the carbonate, glucuronic acid was increased for 2 days, ethylsulfuric acid for 3 days. All but a small amt, of the substance is recovered unchanged in the bowels. With guaiacol cinnamate the secretion of both acids is greatest on the day of administration; on the following day glucuronic acid falls below normal but returns again on the 3rd day when the proportion of both becomes normal. There is also evidence that a third guaiacol-containing substance as yet unknown is secreted. K guaiacolsulfonate increases the secretions but slightly even in large amts. and on successive days. Guaiacol is detd. by distilling an acid soln. with steam and titrating the distillate with o.1 N p-nitrodiazobenzene. Free guaiacol may be detd. in urin by acidulating and titrating in presence of NaAcO. The amt. of glucuronic acid secreted by a normal man was found by Tollens to be 0.35-0.5 g. daily; the author found 1.0 g. Tollens filtered and washed the Pb ppt. after letting it stand 7 hrs., but 24 hrs. are needed. The urin is pptd. with PbOAc and NH, and washed and distilled with HCl 1.06, when the glucuronic acid is decomposed into furfural, CO<sub>2</sub>, and H<sub>2</sub>O; the furfural is pptd. with phloroglucinol and from this the glucuronic acid detd. H. C. FULLER.

Quantitative Estimation of Anthracotic Lung Pigments. W. Boer. Rudolf. Virchow. Krankenhaus, Berlin. Arch. Hyg., 7, 73-9.—The method is based upon dissolving a measured vol. of lung tissue in antiformin which gives a colorless soln. of erganic pigments, leaving anthracotic pigment as ppt. It is applicable to isolation of other inorganic deposits.

G. R. Henry.

Specificity of Reaction of Urin with Liquor Bellostii in Paresis. F. STERN. Univ. Kiel. Münch. med. Wochschr., 58, 467-8.—The author concludes that the reaction is not specific.

G. R. Henry.

Isolation of Copper from Urin and Sweat of a Brass Worker. E. H. GOODMAN. Philadelphia. Manch. med. Wochschr., 58, 624-5.—Copper was detected in urin and in green sweat stains on patient's shirt.

G. R. HENRY.

A New Method for Detection of Salvarsan. J. ABELIN. Bern. Munch. med. Wockschw., 58, 1002-3.—Urin is acidified with dil. HCl, treated with 1-2% NaNO<sub>2</sub> soln., and few drops poured into a 10% resorcinol soln. made alk. by Na<sub>2</sub>CO<sub>2</sub>. A red color is obtained.

G. R. Henry.

Contributions to the Colloid-chemical Analysis of Absorption and Secretion Processes. Absorption from the Abdominal Cavity. M. H. FISCHER. Kolloidchem., 2, 304-42; thru Chem. Zentr., 1911, I, 1873-4.—In accordance with previous work (cf. C. A., 4, 3243-4), absorption and secretion processes in higher animals are considered from a colloid-chemical standpoint. There is no essential difference between absorption in one-celled and many-celled organisms except that the former are surrounded on all sides by the same medium, whereas the absorption and secretion organs of the latter are in contact with different media in different parts. Hence in one-celled individuals absorption and secretion indicate merely the tendency to attain equil. with the surrounding medium, while among many-celled individuals equil. must be established with 2 or more media. Hence among the higher organisms certain cells are chiefly absorptive in their functions, while others secrete. Living and dead guinea pigs were injected intraperitoneally with water heated to 38° and a number of solns. of salts and organic substitutes. The liquids unabsorbed after a definit time were removed and measured. The absorption of the solvent was identical with the absorption of water by a liquid colloid under similar conditions. The absorption from the abdominal cavity thus corresponds completely to that from the intestinal tract, and both are "mirror images" of the secretion from the kidneys. Lymph formation runs parallel to the secretion of urin. The selective character of the absorption and secretion of dissolved substances depends on the unequal partition of the dissolved substance among the 3 phases (H<sub>2</sub>O, cell-structure, blood) of which the absorption and secretion systems of higher animals consist. The phenomena involved can be explained on the same basis as the inequalities observed in the partition of dissolved substances between H.O. a solid and a liquid colloid. The chief theories of absorption and secretion are discussed. The idea that osmotic forces influence the absorption of water should be abandoned. The expts. adduced to show that absorption and secretion are physiològical, selective, or vitalistic phenomena can also be explained by the theory of the formation of colloids with water. M. HEIDELBERGER.

The Action of Combinations of Drugs. EMIL BÜRGI. Med. Chem. und pharm. Inst., Univ. Bern. Z. exp. Path. Ther., 8, 523-35; thru Chem. Zentr., 1911, I, 905-6.

—Earlier communications (cf. Hanckold, Ibid., 7, 743; Lindemann, 7, 725; Hammerschmidt, 8, 374) and those in following abstrs. are based upon the following laws deduced by the author. Drugs which on the whole have similar pharmacological

effects will produce, in combination, effects greater than the additive action of the substances of the mixt. only when the substances have different pharmacological points of attack. Drugs with the same point of attack show in combination a simple additive effect. These laws are confirmed by the behavior of the narcotics of the fatty series, by the opium alkaloids and by the combinations of the aliphatic narcotics with these alkaloids or with scopolamine. The summation of the effects of combinations of antipyretics with aliphatic drugs presents only an apparent exception, since with the former as well as with the latter the narcotic action is based on the same pharmacological effect, viz., soln. of lipoids.

Walter A. Jacobs.

The Mutual Pharmacological Influence of Two Narcotics of the Aliphatic Series on Intravenous Injection. ALEX. SARADSCHIAN. Med. chem. und pharm. Inst., Univ. Bern. Z. exp. Path., 8, 536-54; thru Chem. Zentr., 1911, I, 906; cf. preceding abstr.—Intravenous injection of the combinations urethane-chloroaldehyde and paraldehydeurethane gave rise at the most to additive effects. Indeed at times the effect was somewhat lower than the sum of the sep. actions.

W. A. JACOBS.

The Effect of Narcotics of the Aliphatic Series when together Injected Subcutaneously. DINA KATZENELSON. Univ. Bern. Z. exp. Path., 8, 555-65; thru Chem. Zentr., 1911, I, 906.

W. A. JACOBS.

The Effect of Antipyretics upon the Action of Narcotic Drugs. Sophie Lomonosow. Univ. Bern. Z. exp. Path., 8, 566-75; thru Chem. Zentr., 1911, I, 906.

W. A. JACOBS.

The Action of Combined Opium Alkaloids. VICTORIE ZEELEN. Univ. Bern. Z. exp. Path., 8, 586-600; thru Chem. Zentr., 1911, I, 907. W. A. JACOBS.

Further Investigations upon the Action of Narcotic Antipyretic Combinations.

ROMAN HERZENBERG. Univ. Bern. Z. exp. Path., 8, 576-85; thru Chem. Zentr.,

1911, I, 906.

W. A. JACOBS.

Studies on the Circulation in Man. IV. The Influence of Oxygen Inhalation on the Circulation in a Case of Cyanosis. G. N. Stewart. H. K. Cushing Lab. Exp. Med., West. Reserve Univ. J. Pharmacol., 2, 477-500.—The blood flow in the hands was increased by O inhalation by 30-70% of the combined flow. A greater % increase was obtained when the initial flow was small than when large. The action of O on the blood flow was not associated with any sensible change in the respiratory movements nor with any certain change in the alveolar CO<sub>3</sub> tension. It was therefore conditioned in some way by the increased O tension, and various possibilities are discussed. O inhalation produced no change in the rate of blood flow in the hands of 2 normal men.

ISRAEL S. KLEINER.

Further Data Relating to the Use of Certain Antimonial Compounds in the Treatment of Experimental Trypanosomiasis. L. G. ROWNTREE AND J. J. ABEL. Pharmacol. Lab., Johns Hopkins Univ. J. Pharmacol., 2, 501-5.—Besides further records of animals treated with the Na salt and the triamide of Sb-thioglycollic acid (C. A., 5, 1618, 3078) there is reported work with a compound in the prep. of which succinic acid, KOH and antimony oxide were combined in the mol. ratio 1:1:0.5. Altho this salt is capable of causing the disappearance of trypanosomes, yet in the vast majority of cases its action is of very short duration. It is irritant locally, more toxic than either of the above-mentioned compds. and much less efficient. ISRAEL S. KLEINER.

Study of the Antiseptic and the Pharmacologic Properties of m-Cresol Acetate. I. Greenwald. Lab. Biol. Chem., Columbia Univ., Coll. Physicians and Surgeons. J. Pharmacol., 2, 513-30.—m-Cresol acetate is a colorless, oily liquid, b. 212° (uncor.), has agreeable odor; d<sub>20</sub> 1.048; insol. in H<sub>2</sub>O and glycerol, but is freely miscible with common org. solvents. By 0.5% HCl and 1.0% Na<sub>2</sub>CO<sub>2</sub> it was gradually

decomposed at 40°, but hydrolysis by dist. water proceeded very slowly. It has very slight coagulating properties on egg-white. m-Cresol acetate is a strong antiseptic agent; in this respect it is as powerful as the free m-cresol, but this property is not due—at least in a peptone medium—to a liberation of m-cresol. When given by mouth to dogs, it is about as toxic as the equiv. amt. of m-cresol. It is probably unaffected in the stomach but is decomposed in the intestin. It is non-corrosive and has very little harmful local effect except in the intestin. In the body m-cresol acetate is partially oxidized to dihydroxytoluenes and m-hydroxybenzoic acid, which are mostly excreted as the conjugate sulfuric and glucuronic acids.

I. S. K.

Action of Senecio Alkaloids and the Causation of the Hepatic Cirrhosis of Cattle (Pictou, Molteno or Winton Disease). A. R. CUSHNY. Pharmacol. Lab., Univ. Coll., London. J. Pharmacol., 2, 531-48.—This disease, which occurs in New Zealand, South Africa and Canada, is popularly believed to be associated with ragwort. Animals (cats, white rats and rabbits) poisoned with senecifoline nitrate and senecifolidine nitrate, alkaloids obtained from Cape Colony ragwort (Senecio latifolius), give symptoms, and post mortem findings, so closely resembling those of the Winton disease that the latter is doubtless chronic poisoning from the Senecio alkaloids. Strangely, extracts of S. jacoboea and S. sylvaticus collected in various places proved non-toxic, altho the former is that to cause the disease in Canada. S. vulgaris prepared in the same way was toxic.

The Application of Liquid Air to Toxicology. E. MAMBLI. Pavia chem. pharm. und tox. univ. inst. Boll. della. soc. med. chir. di Pavia, Dec. 3, 1910; thru Chem. Zentr., 1911, I, 1614.—Liquid air is of valuable assistance in preserving portions of cadavers for analysis, since at —190° putrefaction does not take place, the tissues do not undergo histological or chem. alteration and volatil or unstable poisons such as HCN, CO, P, do not escape or decompose: It possesses the additional advantage over other preservatives of lessening the possibility of introducing foreign substances as impurities. The tissues are rendered brittle by the treatment so that disintegration can be effected in a mortar. The high price of liquid air seems to be the only disadvantage accompanying its use.

Inhibition of Invertin Action. ANSELM ERIKSSON. Med. chem. Inst., Upsala. Z. physiol. Chem., 72, 313-38.—Invertin action like some other enzyme action is inhibited by charcoal and serum. It is important that the enzyme be first mixed with the charcoal or serum before addition to the sugar soln. This is however not so important in the case of serum. The extent of inhibition increases with the length of time the enzyme and charcoal or serum are allowed to interact. Inhibitory substances are also present in the inversion soln. These are to a certain degree preformed in the enzyme and are not produced by the heating of the enzyme. They are not affected by heating to 100°, are partly adsorbed by charcoal and diffuse but slowly.

G. M. MEYER.

An Animal Alkaloid. N. D. AVERKEEV. Bergbauschule, Yekatermoslav, Russia. Z. physiol. Chem., 72, 347-62.—Milk was sterilized in a Papin digestor at 102° and put aside at room temp. for 3 years under 4 conditions. (1) Exposed to light and air (cotton plug); (2) exposed to air and protected from light; (3) exposed to light and protected from air; (4) protected from light and air. A sample of milk was analyzed before and after preservation. With (1) there was a marked diminution in the fat (6.5-0.89%) and albumin (2.88-0.86%). The milk had a sharp, unpleasant taste which disappeared on b. There was an increase in acidity for 100 cc. milk from 38 cc, to 42 cc. 0.1 N NaOH. In (2) the milk had a slight reddish tinge, there was a fairly marked diminution in the fat, an increase in the albumin (to 3.02%), and the acidity rose to 50 cc. 0.1 N alkali. In (3) there was a brown color and a strong odor

of rancid fat. There was about 0.87% fat; albumin increased to 3.41%; acidity increased to 50 cc. 0.1 N alkali. When (1), (2) and (3) were neutralized with 10% alkali and distilled with steam into ether, the vacuum distillate from the ethereal layer gave an alkaloid,  $C_{10}H_{10}NO_{10}$  of m. p. 38°, subliming at 52°, sol. in ether, petrol ether, CHCl<sub>2</sub>, watery alc., insol. in benzene and benzine. 700 cc. milk yielded 0.519 g. substance. It was also obtained by the Stas-Otto method. When 0.005-0.008 g. was injected into guinea pigs death resulted in 2-3 hrs. 0.0124-0.031 g. is fatal immediately. When the preserved milk is taken internally it gives rise to nausea and diarrhea. Samples (4) which had been kept 5 years, were nontoxic and did not yield the alkaloid.

Protein Determination and Peptic Digestion. F. WESTHAUSSER. Lab. Landw. Versuchsta., Hohenheim. Z. physiol. Chem., 72, 363-73.—The detn. of protein N according to Stutzer and Barnstein (pptn. with CuSO<sub>4</sub> and NaOH) and the tannin method (detn. of the amide N in the tannin ppt. and protein N by diff.) agree very well provided the product under exam. was at no time subjected to peptic digestion. Discrepancies of the 2 methods were noted, particularly in the protein N detn. of feces.

Extractives of Ox Kidneys. K. Bebeshin. Med. chem. Lab., Univ. Moskow. Z. physiol. Chem., 72, 380-6.—Betaine was obtained from that ext. of the kidneys which corresponds to the one in which Gulewitch and Krimberg isolated carnitine from muscle. Carnosine, methylguanidine, and carnitine are absent in the kidney of the ox.

G. M. MEYER.

Qualitative Determination of Mercury in Urin. E. Salkowski. Path. Inst., Berlin. Z. physiol. Chem., 72, 387-401.—The following method was found to give the most satisfactory results: (1) Evap. the urin slightly acidified with 2 cc. 25% HCl to 500 cc. urin. (2) Oxidize with HCl and HClO<sub>2</sub>. (3) Extract with alc., evap. extract and take up residue with 40 cc. abs. alc. to which 60 cc. ether is added. The ether-alc. ext. is filtered and evapd. Take up residue with 10 cc. H<sub>2</sub>O and filter. This filtrate is tested for Hg by the addition of 5 drops conc. HCl and SnCl<sub>2</sub>. The conversion into HgI has also proved a convenient and reliable test. G. M. M.

Deamination in the Blood. A. K. MEDVYEDEV. Physiol. chem Lab., Univ. Odessa. Z. physiol. Chem., 72, 410-49.—The NH<sub>2</sub> content of the blood of normal, starving and thyro-parathyroidectomized dogs was detd. immediately after withdrawal of the blood and at intervals during its aseptic preservation, by distillation in vacuo into o. 1 N acid with constant addition of alc. to prevent feaming. Lackmoidmalachite green was used as indicator and found to be more sensitive than alizarin. A special app. was devized for the distillation. The av. NH, content of the blood of (a) normal dogs was found at 0.56%, (b) of thyro-parathyroidectomized dogs at 0.79% when the blood was taken during tetany. Prior to death it was higher. (c) Starving dogs 1.80 %. When the blood was collected in oxalate and preserved, the NH<sub>2</sub> content of (a) rose slowly at first and with increasing rapidity reaches a constant. In (b) the rate of increase from the beginning was rapid and also reaches a constant. The NH<sub>a</sub> content of (c) diminished. The general formula  $(dx/dt) = (k_a - k_t)(a - x)$ represents the change in all 3 cases, which resemble that of an autocatalysis. The increase of NH, on allowing (a) and (b) to stand is due to the presence of a deaminase. In (a) the deaminase diffuses out slowly from the blood form elements, with (b) it had already been set free in vivo. The curve of NH<sub>2</sub> increase of (b) can be obtained with the blood of (a) if the same is hemolyzed. G. M. MEYER.

Bumblebee Wax. III. Are Psyll Alcohol and the Alcohol from the Bumblebee Wax Identical? Ernst Edw. Sundwik. Physiol. Chem. Inst., Helsingfors. Z. physiol. Chem., 72, 455-8.—These alcohols are not identical. G. M. MEYER.

Examination of the Blood of Ascidia. I. The Vanadium Compound of the Blood Corpuscles. M. Henze. Chem. Physiol. Dept., Zool. Station, Naples. Z. physiol. Chem., 72, 494-501.—The colorless blood of Phallusia mammalata on standing, first turns brown and then greenish blue. It does not coagulate and like the blood of other Ascidia is acid to litmus. Corpuscles in particular react strongly acid. The chromogen is contained only in the corpuscles. The chromogen was extracted with water and pptd. with acetone. With pyrogallol, pyrocatechol or tannin color changes are brought about. V could be detected in the residue obtained by incinerating the chromogen. A quant. detn. showed the presence of more than 15% Vd<sub>2</sub>O<sub>4</sub> in the chromogen.

G. M. MEYER.

The Physiological Cleavage of Acids and the Synthesis of an Amino Acid in the Animal Organism. Notice on the Work of F. Knoop. D. Ackerman. Z. physiol. Chem., 72, 504; cf. Knoop, C. A., 5, 1449.—An acknowledgment of error.

G. M. MEYER.

New Procedure for Determination of Hydrochloric Acid in the Gastric Juice. JUAN MANUEL NORIEGA. Anales inst. med. nacional, 11, 1-8.—The following procedure is proposed for the detn. of free, combined, weakly combined, and total HCl. (1) Take a 10 cc. sample and neutralize the acidity with Na<sub>2</sub>CO<sub>2</sub>, thus transforming the free and weakly combined HCl into NaCl. Organic matter is then destroyed by Charpantier's method; to the 10 cc. of gastric juice add 3 drops H,SO4, then KMnO4 in excess and warm gently. The excess of KMnO4 is reduced with alc. and the liquid filtered: NaCl is detd, in the filtrate and HCl corresponding is calculated. The no. obtained represents total HCl(A). (2) A 10 cc. sample of gastric juice is evapd. to dryness and the residue ignited in order to destroy organic matter and to eliminate free and weakly combined HCl. Chlorides are detd. in the soln. of the residue by means of AgNO, and correspond to the combined HCl(B). A-B then represents the free plus the weakly combined HCl (C). (3) 25 cc. of gastric juice are distilled in an air-tight app. The gastric juice should be evapd, to dryness without any ignition of the residue; fatty acids and free HCl are thus vaporized, leaving the weakly combined HCl in the residue. The vol. of the distillate is completed to 25 cc. and a 10 cc. sample taken; Na<sub>2</sub>CO<sub>2</sub> is added, organic matter (fatty acids) destroyed, and chlorides detd. as in the preceding cases. The calculated acid represents free HCl (D). C minus D then yields the amt. of weakly combined HCl(E). H. S. PAINE.

Determination of Sodium Chloride in Urin when Bromides or Iodides are also Present. J. M. NORIEGA. Anales inst. med. nacional, 11, 77-9.—The following method is sufficiently rapid for clinical purposes: In a graduated glass tube provided with a rubber stopper, take 10 cc. of urin and an equal vol. of HCl, then 3 cc. of H<sub>2</sub>O<sub>2</sub> and finally 10 cc. of CHCl<sub>2</sub>. The mixt. is shaken (taking care not to emulsify the CHCl<sub>2</sub> and urin until all the free Br or I is dissolved in the CHCl<sub>2</sub>). The I or Br content is then detd. colorimetrically by comparison with a soln. composed of 10 cc. distilled water (containing 0.01 g. iodide or bromide of Na or K, according to which salt was found present by preliminary test) and HCl, H<sub>2</sub>O<sub>2</sub>, and CHCl<sub>2</sub>, as above, the necessary dilns. being made with CHCl<sub>2</sub>. NaCl may then be detd. as usual by AgNO<sub>3</sub>, correction being made for iodides or bromides as detd. by the method above (with necessary regard to the mol. wts. used in the calculation).

H. S. Paine.

Determination of Urea by a Cryoscopic Method. ACHILLES MACHADO. J. sci. math. phys. naturaes, [2] 7, 129-33.—Employing the usual equation for the reaction between urea, NaBrO, and NaOH in the ordinary method for detg. urea and taking into account the degree of dissociation of the electrolytes present before and after the reaction, it is seen that the no. of mols. and ions is less after the reaction is completed by 2.4 for every mol. of urea originally present. If the f. p. of H<sub>2</sub>O is lowered

1.85° for every g. mol. present in 1 l., then for every 0.01 g. of urea in a 20 cc. vol. the difference in depression of the f. p. of  $H_2O$  before and after the reaction would be  $2.4 \times 1.85 \times 50/6000 = 0.037^\circ$ . A Beckmann app. and differential thermometer are used in the detn. Actual measurements with the NaBrO reagent (composed of 100 g. NaOH soln. of d. 1.33, 200 cc. of dist.  $H_2O$ , and 10 cc. Br) and a soln. of pure urea showed a change in depression of the f. p. before and after the reaction of 0.045° instead of 0.037° per cg. of urea in 20 cc. vol. originally present and this figure was adopted for the calc. The depression of the f. p. of the mixed reagent and urea soln. (without chem. reaction) can be detd. by adding the depressions for the 2 solns. measured separately. In this way the method can be applied to the detn. of urea in urin. An error (causing high results) may be introduced thru the neutralization of the acidity of the urin by the alkali of the reagent; this can be obviated by making the urin slightly alkaline before detg. its f. p. The NaBrO reagent must be protected from  $CO_2$  of the air.

Antagonism between Magnesium, Calcium and Barium. J. Gómez Ocana. Madrid. Rev. real acad. cien., Madrid, 9, 591-634.—By injection of BaCl<sub>2</sub> soln. in one animal. a Mg salt in a 2nd, and both BaCl<sub>2</sub> and the Mg salt in a 3rd (rabbits were used), it was ascertained that BaCl<sub>2</sub>, tho exerting by itself an initial convulsive motor effect terminating in motor paralysis, opposes and neutralizes the paralytic and anesthetic action of Mg salts. For complete neutralization of the Mg effect the Ba salt must be injected previous to the Mg injection (30 or 40 min. in this case); otherwise (and also in case of insufficient amt. of Ba salt) the toxic symptoms of Mg are only diminished in varying degree. A similar antagonistic action between Ca and Mg was also found.

H. S. Paine.

Biological Action of Calcium and Magnesium. José Gómez Ocana. Rev. real acad. cien., Madrid, 8, 947-75.—Ca salts in small amts, have a favorable influence on the excitation of muscle fiber; this is in accord with the small % of Ca normally present in the tissues and with the necessity of a certain conc. of Ca ion for the normal functioning of the protoplasm, especially in nerve and muscle. Expts. on injecting Mg salts confirmed most previous work on the subject. The curare-like action of Mg salts should rather be described as anesthetic. The local interruption of conduction in nerves by Mg salts in conc. soln. (observed by Meltzer and Delhaye) was not confirmed. The antagonistic action of Ca and Mg was shown by the re-establishment after injection of CaCl, soln., of muscle contractions which had been suspended after the injection of Mg salts.

Hormone Affecting the Mammary Glands in Pregnancy. ARTUR BIEDL AND ROBERT KÖNIGSTEIN. Z. exp. Path., 8, 358-73; thru Chem. Zentr., 1911, I, 167.— Intraperitoneal implantation of a placenta containing embryos caused the mammary glands to undergo the characteristic changes. This is taken as confirming the view that a hormone is developed in the embryo and comes thru the placenta into the blood and thence to the mammary glands.

I. K. Phelps.

Action of the Salts of Alkaline Metals on the Conversion of Starch to Sugar by Amylolytic Enzymes. VII. Ammoniacal Salts of Organic Acids. C. Gerber. Compt. rend. soc. biol., 71, 41-3; cf. C. A., 5, 2396.—Neutral ammoniacal salts of organic acids, independently of both the acid and of the amylolytic diastase, act like the salts of mineral acids in the conversion of starch to sugar by amylolytic enzymes. In small amts. they accelerate the action, retard in large amts., and check completely in still larger amts. Even NH<sub>4</sub> oxalate is no exception to this rule. Di-NH<sub>4</sub> citrate behaves like mono-K and Na salts. Large amts. of the citrates appear to destroy the diastase. Tablesa re given in illustration. VIII. Salts of Amines. Ibid., 43-5.—Primary amines behave like the NH<sub>4</sub> salts. Secondary and tertiary amines behave

similarly, altho a given quantity of these amines does not check to the same extent as an equal quantity of the primary amines. IX. Amides and Nitriles. 1bid., 45-7.—Acetamide retards the conversion of starch to sugar by amylolytic enzymes only in large amts., accelerating in all other proportions and increasingly accelerating according to the amount of acetamide added. Acetonitrile has little or no action when present in smaller amts. and checks completely in larger amts. The action of the acetonitrile is due to its pptn. of both starch and diastase. Urea has little action in very small amts., retards in larger amts., and checks completely in still larger amts.

I. K. PHELPS.

Some Reactions Given by Phenolphthalein in the Identification of Blood. A. SARTORY. Compt. rend. soc. biol., 70, 965-7.—A record of results of testing Meyer's reagent in the presence of various salts, both organic and inorganic, together with some organic substances other than salts, and saliva and blood.

I. K. Phelps.

Traces of Cholesterol in Normal Urin. ER. GERARD. Compt. rend. soc. biol., 70, 998-1000.—Details of the extraction of 10 l. of normal urin for cholesterol are given. Cholesterol is normally present in small amts.

I. K. Phelps.

General Existence and Reappearance of Antithrombin in the Organism. M. DOYON AND A. POLICARD. Lab. Physiol. Fac. méd. Lyon. Compt. rend. soc. biol., 71, 8-9; cf. C. A., 5, 2116.—The authors find antithrombin in other organs than the liver and believe it may be found in all. It is found by heating 40 min. in an autoclave at 120°, in a weakly alkaline soln., expressing the juice, and pptg. the phosphoprotein by HOAc.

I. K. Phelps.

Influence of Oxidation on the Toxicity of Urohypotensin. J.-E. ABELOUS AND E. BARDIER. Lab. physiol. Fac. méd. Toulouse. Compt. rend. soc. biol., 71, 62-3.—Urohypotensin heated at 40-5° for 2-3 hrs. with an equal wt. of NaClO<sub>2</sub> gave material much more toxic than urohypotensin. This is supposedly due to the formation of nitriles.

I. K. Phelps.

Arsenic in the Milk of a Goat Injected Intravenously with Salvarsan. CHAMBRELENT AND CHEVRIER. Compt. rend. soc. biol., 71, 136-8.—The milk of a goat injected with salvarsan was found free from As.

I. K. PHELPS.

Cholesterol and Oxycholesterol in the Serum in Different Forms of Insanity. Giacomo Pighini. Z. Neurol. Psych., 4, 629-44; thru Zentr. Biochem. Biophys., 11, 738.—The cholesterol and oxycholesterol found in imbecility, senil dimentia, and in most cases of epilepsy was normal. Comparatively high values were found in maniac depressive insanity and dementia praecox. A distinct parallelism was found between the content of cholesterol and the acute stages of alcoholic insanity. Oxycholesterol was also high in these cases. High values were found in most cases of progressive paralysis.

I. K. Phelips.

Viscosity and Diastatic Reactions. Hypothesis Concerning the Nature of Diastases. P. ACHALME. Compt. rend., 152, 1621-4.—Theoretical discussion of the nature of enzyme action. The author considers an enzyme in colloidal soln. as composed of granules undergoing Brownian movements, and thus losing energy as electrons or vibrations in the ether. When the velocity of the electrons or the period of vibration corresponds with the intramolecular vibrations of a substance which is capable of being affected by the enzyme, a phenomenon comparable to resonance occurs, which has its analog in the photochemical actions of ultraviolet light. The specificity of enzymic action depends upon the relation between the radiations and the substance.

H. A. SPORHR.

Determination of Amino Groups in Amino Compounds and in Urin and a Method for the Analysis of Proteins. DONALD D. VAN SLYKE, Lab. Rockefeller Inst. Med.

Research, New York. Ber., 44, 1684-92; see C. A., 5, 509.—In the author's app., aminoacetic acid and cystine evolve 103 and 107%, resp., of the calculated quantity of gas, not absorbed by "permanganate" soln. Lysine requires 30 instead of 5 mins. for its decomp. Cytidine and adenosine evolve 1 mol. of N, guanosine about 1.25 mol. and guanine, on account of its insol. in dil. acids, does not react completely even after many hours. The purines, NH, and MeNH, require about 2 hrs. Shaking should be done at the end of reaction time, so as to bring the gas and liquid into equil. A blank test should be made. Carbamide, as is well known, reacts only slowly with HNO. The urin is treated with aq. NaOH until the mixt. contains 40% of NaOH, the NH, is displaced by air, the residue acidified with AcOH and evapd. to 0.5 its bulk. The NH, N is then detd. separately in 2 portions (A) for 6 mins. and (B) for 12 mins. Each mixt. is shaken during the last min. of the resp. times. The difference in the quantity of N evolved by (A) and (B) represents the amt. of N evolved by the wea in 6 mins. If this difference be subtracted from (A), the remainder gives the N from the NH<sub>2</sub> groups. An improved scheme is described for the sepn. of hydrolytic products of proteins. It is illustrated with products obtained by the hydrolysis of fibrin. J. BISHOP TINGLE.

Two Indoxyl Reactions and a New Red Urin Coloring Matter. Derregin. Chem. Ztg., 34, 925.—If one replaces the isatin in the Baeyer-Bourna reaction with sulfoisatic acids one obtains a red coloration. By using methyl-4-hydroxy-2-furfural instead of furfural in Nicolas' method one obtains a red coloration.

E. J. WITZEMANN.

Translocation of Plant Food and Elaboration of Organic Plant Material in Wheat Seedlings. J. A. Le Clerc and J. F. Breazeale. U. S. Dept. of Agr., Bur, Chem., Bull. 138.—The authors sprouted wheat on perforated disks of Al, floated in shallow pans of water, and detd. the movement of both the organic and inorganic salts from the bran and endosperm into the embryo. The movement of K was especially active. When the plumule and radicle were only 2 days old and weighed only about 6% of the whole seed, they contained about 50% of the K of the original seed as compared with 25% of the N and 17% of the H,PO4. There was no exchange of fat from the bran and endosperm into the sprouting embryo. The residual seed after sustaining the plantlet for 17 days contained as much fat as was found in the bran, alleurom, and endosperm of the original seed. There was a steady increase in the amt. of fiber in the growing seedling, while the amt. of sugar in both the plant and residual seed increased steadily until the 7th or 9th day, when it began to decrease. Plants were also grown in solns. containing nitrates, phosphates and potash, and the effect of these salts upon the movement of the plant foods to the seedling detd. The movement did not seem to be materially affected by an outside application of these salts.

J. F. BREAZEALE.

Atriplex halimus L. G. PARIS. Staz. sper. agrar. ital., 44, 141-56; thru Chem. Zentr., 1911, II, 35.—A botanical description of the plant. Results from analyses of the leaves and their ash, of the fruit stems and their ash, and from the detn. of NaCl and oxalic acid in the individual organs, are given in tabular form.

J. A. LECLERC.

Properties of Several Species of the Polyporaceae and of a New Variety of Clitocybe (Clitocybe dealbata sudorifica, Peck). W. W. FORD AND J. L. SHERRICK. Lab. Hyg. and Bacteriol., Johns Hopkins Univ. J. Pharmacol., 2, 549-58.—Bolletus felleus was free from agglutinins and muscarine but produced in guinea pigs and rabbits a chronic intoxication which was eventually fatal. B. miniato-olivaceus contained a thermostabil agglutinin but no muscarine. It was poisonous to guinea pigs but not to rabbits. B. chromapes was free from agglutinin and muscarine. It was poison-

ous to guinea pigs but not to rabbits. B. affinis, B. ornatipes, B. bicolor and B. separans contained agglutinins but no muscarine; they were non-toxic. B. ravanelis contained no agglutinin or muscarine and was non-toxic. B. ravanel contained an agglutinin but no muscarine. It was toxic to a rabbit but not to guinea pigs. Strobilomyces strobilaceus contained no agglutinin or muscarine and was non-toxic. No hemolytic substances were found in any of these species. The new Clitocybe dealbata sudorifica has been found to cause intense perspiration, lasting several hrs. When tested upon rabbits, guinea pigs, and the exposed frog heart a watery extract of this fungus produced reactions characteristic of the muscarine-pilocarpine series, indicating the probable presence of muscarine.

ISRAEL S. KLEINER.

Role of Mineral Matter in the Formation of Protease of the Anthrax Bacillus. Jean Bielecki. Compt. rend., 152, 1875-7.—The anthrax bacillus develops in soln. of aspartic acid, which soln. then possesses proteolytic properties. The addition of 1 drop N soln. of various salts per 1. considerably influences the formation of the protease or of the gelatinolytic ferment. Ca (1 drop satd. soln. Ca(OH)<sub>2</sub>), accelerates the gelatinolytic power, whereas K, Na, NH<sub>2</sub>, or Mg, diminish this action. The reverse is the case in regard to the proteolytic ferment.

G. M. Meyer.

Contribution to the Knowledge of Yeast Gum. H. EULER AND A. FODER. Biochem. Lab., Hochschule, Stockholm. Z. physiol. Chem., 72, 339-46.—Yeast gum was hydrolyzed and the mannose and glucose detd. quantitatively. The results indicate that the gum might be a complex of either 4 mannose and 3 glucose  $[(C_0H_{10}O_b)_{70} + xH_2O]$ , or 4 mannose and 4 glucose  $[(C_0H_{10}O_b)_{20} + xH_2O]$ , molecules. G. M. M.

The Localization of Betaine in Plants. V. STANEK. Versuchsta. für Zuckerind., Prag. Z. physiol. Chem., 72, 402-9.—The distribution of betaine in plants is very irregular. The leaves usually contain the most. The early spring leaves are richer in betaine than those of the fall. The bark of Lycium and Atriplex has but little and the root of Amaranthus has only 0.48% as against 2.16% in the leaves. The seeds contain betain but sparingly.

G. M. MEYER.

The Reduction of Furfural by Yeast during Alcoholic Fermentation. C. J. LINTNER AND H. H. V. LIEBIG. Gärungschem. Lab., Techn. Hochsch., Munich. Z. physiol. Chem., 72, 449-54.—Furfural is reduced by yeast to furfuryl alcohol. Besides this a cryst. substance was obtained from the fraction b. at 200-40°. The identity of this compd. could not be established. It m. at 50.5° and distils with partial decomp. at 235°<sub>122</sub>. It is very sol. in water and volatilizes as does camphor when exposed to the air.

G. M. MEYER.

Report on Inorganic Plant Constituents. O. M. Shedd. U. S. Dept. Agr., Bur. Chem., Bull. 137, 30.—The methods studied during the year were the peroxide method for S in plants and a method for sepg. Fe<sub>2</sub>O<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> in ash. Graham flour and linseed meal gave good results for S, and it is recommended that the method be made official. The only changes in the previous directions are to expel the Cl soln. before or during the pptn. with BaCl<sub>3</sub>, and to allow to stand for 24 hrs. as the large amt. of NaCl in the soln. retards the pptn. For sepg. Fe<sub>2</sub>O<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub>, the H<sub>3</sub>PO<sub>4</sub> is pptd. by NH<sub>4</sub> molybdate at a temp. not exceeding 40° C. Otherwise no change. Other methods are recommended for study.

The Function of Hormones in Regulating Metabolism. H. F. Armstrong and E. F. Armstrong. Ann. Botany, 25, 507-19.

H. A. Sporhr.

The Role of Nucleoproteins in Plants. W. Zaleski. Lab. of Plant Physiology, Cracow. Ber. Botan. Ges., 29, 146-55.—The amt. of nucleoprotein P<sub>2</sub>O<sub>3</sub> was detd. in different parts of various plants and under various conditions and stages of development. Nucleoproteins are formed in all growing cells. As these substances do not

decrease in amt. during inanition, they are to be regarded primarily as formative material which play an important part in the formation of protoplasts and in other constructive processes.

H. A. Spoehr.

The Chemical Composition of the Plasma Membrane. W. W. LEPESCHKIN. Kasan. Ber, botan, Ges., 29, 247-61.—A theoretical discussion of the modern views of the plasma membrane and particularly of the theory which states that in order that a certain substance may pass thru the plasma membrane it must be sol, therein, is given. Upon this theory the expts. are founded. The presence of proteins in the plasma is established by the comparison of the conditions under which the plasma membrane of various plants (Spirogyra crassa, Tradescantia discolor, Saccharomyce's cerevisiae) and egg albumin coagulate. Thus it was found that the conc. of MeOH, EtOH, AcH, Me.CO, amd chloral hydrate necessary to bring about coagulation was the same for the plasma and the albumin. Similarly the conc. of EtOH which reduces the temp. at which coagulation takes place was relatively the same, e. g., for Tradescantia and egg albumin, resp.; without EtOH coagulation takes place at 72° and 61.2°, in 4% EtOH at 65.7°, 56.3°; in 8% at 58.5°, 53.7°; in 16% at 43.9°, 46.7°. The presence of lipoids in the plasma was established by means of the solubility of anesthetics in these substances. This if Spirogyra is placed in an aq. soln. of ether, the amt. of this substance passing into the plasma, by dissolving in the lipoids, is proportional to the partition coeff. between H.O and the lipoid, and in turn proportional to the conc. of the aq. ether soln. Furthermore the presence of a certain conc. of an anesthetic in the plasma causes the coagulation of the proteins therein. With various plants such a coagulation took place when the plants were in aq. solns. of anesthetics which were 2.6-41 times too dil, to cause a coagulation of egg albumin. The plasma membrane therefore contains lipoids which can selectively dissolve sufficient anesthetic from the aq. soln. to cause coagulation of the proteins. H. A. SPOEHR.

A Propos of a Recent Communication of L. Caillet. L. MAQUENNE. Compt. rend., 152, 1818-9.—In connection with the recent work of Caillet (C. A., 5, 2664) who concluded that certain shade plants gain their C from the soil, the author shows that Aspidistra, the plant used by Caillet, in particularly somber weather decomposed 0.03 cc. CO<sub>2</sub> per sq. cm. of surface which is sufficient to cover respiration. It is necessary to measure the conditions of light and temp. under which the expts. of Caillet were carried out in order to avoid ambiguity.

H. A. Spoehr.

The Mechanism of Alcoholic Fermentation. A. Lebedev. Compt. rend., 153, 136-8.—With yeast juice prepared according to Lebedev's process, dihydroxyacetone (in conc. of 5 to 100) ferments as easily as cane sugar. In a mixt. of 60 cc. of juice, 3 g. dihydroxyacetone, 2 g. Na<sub>2</sub>HPO<sub>4</sub> and 1 g. NaH<sub>2</sub>PO<sub>4</sub>, 0.6615 g. P<sub>2</sub>O<sub>5</sub> entered into organic combination after 1 hr. and 25 min., when the fermentation was interrupted, and by means of phenylhydrazine the osazone,  $C_{21}H_{21}N_{4}O_{7}P$ , was obtained; the Ba salt,  $C_{2}H_{10}O_{4}(PO_{4}Ba)_{2}$ , obtained from the fermentation of cane sugar. This osazone is the intermediate product in alc. fermentation of all sugars. The following equations gave the reactions involved:  $C_{2}H_{12}O_{6} = 2C_{2}H_{6}O_{2}$ ;  $2C_{2}H_{6}O_{2} + 2RHPO_{4} = 2C_{2}H_{5}O_{2}RPO_{4} + 2H_{2}O$ ;  $2C_{2}H_{5}O_{2}RPO_{4} = C_{6}H_{10}O_{4}(RPO_{4})_{2}$ ;  $C_{6}H_{10}O_{4}(RPO_{4})_{2} + H_{2}O = C_{2}H_{5}OH + CO_{2} + C_{2}H_{5}O_{2}RPO_{4} + RHPO_{4}$ .

H. A. Spoehr.

Lipase Produced by Bacteria. H. L. SÖHNGEN. K. Akad. Wetenschappen, 1911, 1263-74; Chem. Zentr., 1911, I, 1708.—The nature of the culture medium is of no importance for the formation of lipase by microörganisms. With sufficient available C and N the organism produces lipase. H ions inhibit and OH ions increase the decomp. of fat; when the culture liquid is about 0.02 N acid the action ceases entirely. Lipase unites with acids to form compds. from which the lipase can again be set free by means of bases. These acid lipases (with the exception of those formed with the

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higher fatty acids) diffuse, as does lipase, thru gelatin and agar. Fats are not decomposed by acid lipases. Ca and Mg ions, trimethylamine, and Na glycocholate favor the action induced by bacterio-lipase; univalent alcs. retard the process, while sugars and glycerol are without effect. O and light favor the action. Fats may be synthesized by the action of bacterio-lipase on oleic acid and glycerol; the monoglyceride is the main product.

H. A. Spoehr.

The Cola-like Masses of the Compositae. F. W. DAFERT AND R. MIKLAUZ. Kais. Kgl. Hoj and Staatdrik. Wien, 1911 (separate).—The comp. of the coal-like masses, called Phytomelanes, varies in the different species. These substances were found to be free from N, very rich in C, and probably formed from cellulose by dehydration.

H. A. Spork.

The Relation between Photosynthesis of Carbon Dioxide and Nitrate Reduction. H. A. Sporhr. Science, 34, 63-4.—As CH<sub>2</sub>O, theoretically the first product of H<sub>2</sub>CO, reduction, is condensed to form sugars in an alkaline soln. only, it is of importance to det. how much alkalinity could be produced in the plant leaf. Ca(NO<sub>2</sub>), and KNO<sub>4</sub> are reduced to NO<sub>2</sub> and NH<sub>3</sub> by means of ultraviolet light, sunlight, and even diffuse light, with the liberation of O and the production of an alkaline soln. Thus not only would the proper conditions for carbohydrate synthesis be established but N w uld also be produced in the best form and most available place for protein synthesis. There follows a theoretical discussion of the problem of H<sub>2</sub>CO<sub>2</sub> reduction in the plant leaf.

H. A. SPORHR.

Action of Dilute Acids on Gelatin (PROCTER). 29.
Allochlorophyll (MARCHLEWSEI, MARSZALEE). 10.
Determination of Mercury in Urin (HEINZELMANN). 7.
Fruit Respiration (GORE). 12.
Inhibition of Growth (STADLER). 14.
Lime and Magnesia in Soil (LEMMERMANN). 15.
Phosphorus in Grapes (VENTRE). 16.
Stovaine (VELEY, SYMES). 17.

HEIM, L.: Lehrbuch der Bakteriologie. 4 Aufl. Stuttgart: F. Enke. 15 M.

## 12. FOODS.

W. D. BIGELOW.

Regulation of the Trade in Foodstuffs in Spain. Royal Order of December 22, 1908. Anon. Z. ôffentl. Chem., 17, 174-80.—Standards are given for: H<sub>2</sub>O; ice; milk and its preps.; butter; cheese; oils, under which it is noted that only olive oil may be sold as edible oil; lard; flours; bread and bakery goods; wines; beer; cider; alcohol, brandies and liquors, under which it is noted that when artificially colored or when containing added essences such liquors must be labeled "colored" or "artificial," these words being in the same type as the name of the liquor; coffee; tea; chocolate and cocoa, under which no more than 4% cocoa shells may be used; syrups, under which it is stated that any artificial syrup whose exact nature is not fully explained to the buyer is considered as adulterated; carbonated waters and drinks; sugars; glucose; invert sugar; honey; vinegar, which shall contain at least 6% CH<sub>2</sub>COOH; salt.

R. F. BACON.

The Condition of Health of the Prussian States for the Year 1909. Anon. Z. Offentl. Chem., 17, 247-56.—Extract from the 4th part of the above report, discussing adulteration of food and drugs found during 1909.

R. F. BACON.

Report on Organic and Inorganic Phosphorus in Foods. H. S. GRINDLEY AND E. L. Ross. Bur. Chem., Bull. 137, 142-4; see C. A., 5, 1952. C. P. WILSON.

Extract from the Official Methods for the Analysis of Wines, Vinegars and Oils by the Laboratories of the State of Portugal. Anon. Ann. fals., 4, 399-403.—Analytical standards are given for wines, vinegars and oils, the last evidently meaning only olive oil.

R. F. BACON.

Food and Medicins of the Ancient Egyptians. F. NETOLITZEY. Z. Nahr. Genussm., 21, 607-13.—An exam. was made of the bowel content of mummies of 3000-4000 B.C. Muscular tissue was not observed but vegetable fibers, grain, bones and scales of fish, and bones and teeth of rodents were observed. Tilapia nilotica, Barilius nilotica and a species of carp all native of the Nile were identified. The remains of rodents were found in the body of a child and it is known that mice were used as a remedy in diseases of children. In one case a woman had eaten 20 different kinds of fish shortly before death.

H. C. FULLER.

The Purity of Foods and Drugs. A. R. TANKARD. Pharm. J., 87, 4-6, 71-3.—
In Hull the av. comp. of milk thruout the year is 3.6% fat and 8.9% non-fatty solids; about 1 sample in 9 is adulterated. A meat preservative was found consisting of K metabisulfite, Na<sub>2</sub>SO<sub>4</sub>, CaSO<sub>4</sub> and powdered glass; ZnSO<sub>4</sub> is sold as cheese spice to prevent the heading and cracking of cheese. Cottonseed and coconut stearins are sold as lard substitutes and are being loaded with H<sub>2</sub>O and paraffin. Whole meal, brown bread and bread made from 80% flour have no advantage in nutritive value over white bread as the proteins are not completely absorbed. Mineral matter used in making bread has been found to contain 0.07 grain As<sub>2</sub>O<sub>2</sub> per 1b. H. C. FULLER.

Report on the Separation of Meat Proteins. R. C. MOULTON. U. S. Dept. Agri., Bur. Chem., Bull. 137, 148.—Difficulty was found in checking Kjeldahl results, using 2 g. samples of beef ext. With 0.5 g. samples lower but more consistent % of N were obtained. This is explained by incomplete action with large samples, giving organic bodies upon distillation that obscure the titration. It is recommended that these cleavage products be studied.

D. S. Pratt.

Comparative Rate of Decomposition in Drawn and Undrawn Poultry. M. E. Pennington. U. S. Dept. Agri., Bur. Chem., Circ. 70.—The circular gives the chem. and bacteriological data from shipments of poultry under carefully regulated conditions. The exam. showed the results of undrawn, wire drawn, Boston drawn, and fully drawn poultry. Poultry was handled and shipped under market conditions and samples were taken at time of shipment and also at the wholesalers and retailers at the beginning and end of the sale of the lot. The data depended on mostly are the acidity of the fat and the NH, and bacterial count of the flesh. When properly handled the full drawn poultry gave the best results in the hands of wholesalers but the worst results at the retailers. Considering all conditions of marketing the undrawn poultry are superior in all respects.

W. D. Bigelow.

Tin Salts in Canned Goods of Low Acid Content, with Special Reference to Canned Shrimp. W. D. BIGELOW AND R. F. BACON. U. S. Dept. Agri., Bur. Chem., Circ. 79.—Many non-acid foods attack the Sn lining of containers to a very marked extent. The ratio of the Sn to acid was detd. in a number of canned foods. Those having the highest ratio were non-acid vegetables, while the acid fruits and vegetables had the lowest ratio. The attack on tin cannot be explained in many cases by the action of an acid. Shrimps contain a corrosive substance which interferes with their hand-

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ling and attacks tin quite vigorously. N(CH<sub>2</sub>)H<sub>2</sub> was obtained from canned shrimp and was found to dissolve Sn readily. Several alk. substances, amino acids, and purine bases were tested in dil. soln. for their action on Sn. Many dissolved considerable quantities. The action of non-acid foods in dissolving Sn is believed to be due to their content in amines and amino acids.

R. F. BACON.

Shellfish Contamination from Sewage-Polluted Waters and from other Sources. G. W. STILES. U. S. Dept. Agri., Bur. Chem., Bull. 136.—A review of the literature regarding the pollution of shellfish, together with a description of typical shellfish beds and packing houses. The bacter ological results of the exam. of samples are given, together with photographs of the establishments.

W. D. BIGELOW.

Are the Phenomena of the So-called Peroxidases Due to a Ferment? GRIMMER. Z. Nahr. Genussm., 21, 676.—Correction to an article by Hesse and Kooper on the above subject (C. A., 5, 2274). The author states that in the serum of raw acid milk, in many cases, a peroxidase reaction is only obtained when the lactic acid is neutralized with Na<sub>2</sub>CO<sub>2</sub> soln., carefully avoiding an excess of this alkali. R. F. BACON.

Investigations on Lactose and By-products of the Lactose Industry. I. A. Burrand F. M. Berberich. Chem. Ztg., 35, 751-2.—The by-products of this industry are milk-albumen, filter press residue and milk sugar molasses. This paper is devoted to analyses of crude lactose. The average max. and min. figures for the products of 11 German factories are resp.: moisture, 2.34, 14.07, 0.25; lactose, 89.60, 95.95, 78.45; albumen, 1.96, 3.22, 0.62; fat, 0.187, 0.415, 0.080; total ash, 2.70, 5.44, 1.23; water insol. ash, 2.06, 4.02, 1.08; water sol. ash, 0.65, 1.42, 0.06; acid as lactic, 0.437, 2.136, 0.068. The averages of 15 analyses of ash from the product of 1 factory were CaO, 30.50; MgO, 4.50; Fe<sub>2</sub>O<sub>3</sub>, 0.55; P<sub>2</sub>O<sub>6</sub>, 3.72; SO<sub>4</sub>, 6.10; Cl, 0.99. P. B. Dunbar.

Tuareg Cheese. G. DE GIRONCOURT. Compt. rend., 153, 191-4.—This cheese, made by the Berber tribes of the middle Niger Valley, is peculiar because of its low H<sub>2</sub>O content (8.0%). Probably because of this it keeps unusually well. The following analysis is given: H<sub>2</sub>O, 8.90%; total N, 38.59; sol. N, 1.77; NH<sub>2</sub>, 0.0; fat, 36.0; ash, 3.9; volatil acid, 0.0; lactic acid, 5.08. The fact that the cheese is high in lactic acid while containing no volatil acids is pointed out.

H. S. Bailey.

A Cheese for Small-Holders and others—the "Kingston" Cheese. ALEC TODD AND WILFRED SADLER. J. Board Agr., 18, 193-203.—Instructions for the manufacture of small cheeses.

G. A. ROUSH.

Cream Ripening. R. PAPR. Agr. J. Union So. Afr., 1, 434-42.—A discussion of the different processes of ripening cream with descriptions of the machines used. Ripening by means of pure cultures at  $10-22^{\circ}$  is recommended. The amount of pure culture added as a "starter" should be 4-10%, depending on the temp. at which the ripening is carried on, the amount being decreased as the temp. increases. G. A. R.

Experiments to Test the Color Table Used with Schaeffer's Whey Tester. O. VON SOBBE. Milchw. Zentr., 7, 172-4; thru Chem. Zentr., 1911, I, 1559.—It was attempted to detn. whether the amts. of Fe<sub>2</sub>O<sub>2</sub> correspond with the color tints given in Schaeffer's color table (C. A., 4, 619). The expts. were carried out exactly according to Schaeffer's directions. It was shown, that after 10 min. an agreement in the colors had not been reached, but developed gradually and was complete after 3 hrs. Since Schaeffer had based his results on an observation time of 10 min. it must be assumed that there is an error in his tables, and the correct percentages of Fe<sub>2</sub>O<sub>2</sub> are not given by them. The tables are correct when one increases the amt. of Fe<sub>2</sub>O<sub>3</sub> represented in his tables five-fold (Klein says 10-fold). If Schaeffer had expressed his results as g. per 20 g. whey, instead of in percentages, his table would be correct.

R. F. BACON.

Schaeffer's Whey Tester. GRIMMER. Milchw. Zentr., 7, 211-3; thru Chem. Zentr., 1911, II, 50:—The color tint obtained with this app. is dependent not only on the amt. of Fe present, but also on the amt. and conc. of the (NH<sub>4</sub>)<sub>2</sub>S. For this reason the usefulness of this method is very doubtful. More sensitive, more convenient and simpler is the NH<sub>4</sub>NCS test, in which some whey is treated with 2-3 cc. H<sub>2</sub>O, 1 cc. HCl, and a few crystals of NH<sub>4</sub>NCS. In the presence of Fe a red color always appears. For the practical man it is sufficient to know whether or not Fe is present in any considerable quantity (cf. preceding abstract).

R. F. BACON.

[Milk and Butter.] A. NESTRELYAEV. Milchw. Zentr., 7, 214-33; Chem. Zentr., 1911, 11, 45.—The fat content of milk (98 samples) averaged 4.55%, 65.31% of the samples containing 4-5% fat. The fat content varied from 3.10-7.56%. Butter (360 samples) contained 12.23% water (8.27-18.66%). The refractometer no. averaged 42.53 (39.5-45.6), the R. M. no. 26.58 (20.92-33.51), the sapon. no. av. 225.36 (215.53-232.25) for the period 1907-1910. The av. values for butter were H<sub>2</sub>O 12.03, ref. no. 42.78, R. M. no. 27.13, sapon. no. 225.54.

W. D. BIGELOW.

The Detection of Nitrates in Milk which has been Treated with Hydrogen Peroxide. VTz. Munich. Z. angew. Chem., 23, 977.—H<sub>2</sub>O<sub>2</sub> as well as nitrates gives a blue color with diphenylamine and H<sub>2</sub>SO<sub>4</sub>. The addition of the catalase hepin to the milk and warming to 35° removes the H<sub>2</sub>O<sub>2</sub> completely.

G. W. STRATTON.

Nature of the Cellular Elements in Milk. III. The Milk of Animals other than the Cow. R. J. Hewlett, S. Villar and C. Revis. British Dairy Farmers' Assoc. J. Hyg., 11, 97-104.—The results confirm the conclusions of the previous papers (*Ibid.*, 10, 91) that a uniform type of life tends to a fairly uniform excretion of tissue cells. High counts are not necessarily associated with disease. Cells of high counts usually show less degeneration than those of low counts.

J. H. MITCHELL.

The City Milk Supply. J. S. McFadzean. J. Dept. Agr. Victoria, 9, 297-311.

—Discussion of the conditions contributing to the production of a sanitary milk supply.

G. A. ROUSH.

The Pasteurization of Separated Milk. C. J. REAKES. J. N. Zeal. Dept. Agr., 360-4.—Proper pasteurization of the milk prevents the spread of tubercular disease to stock to which it is fed. Pasteurized milk is a better food than milk not pasteurized.

Cleanliness in Connection with Milking Machines. M. A. O'CALLAHAN. Agr. Gaz. N. S. W., 22, 506.

G. A. ROUSH.

The Handling of Milk According to the Most Recent Chemical Investigations. DIETZE. Milch Ztg., 40, 167-8; thru Chem. Zentr., 1911, I, 1709.—Since raw milk contains bactericides which are destroyed by heat, it is more healthful than sterilized milk, provided it is obtained clean, from healthy animals. These bactericides which normally disappear in a short time may be retained active for 24 hrs. by the perhydrate method of Much and Römer, which is briefly described.

H. S. Bailey.

Sheep's Milk in the Adulteration of Cow's Milk. A. Funaro and L. Musante. City Lab., Livourne. Ann. fals., 4, 403-5.—As the d. of sheep's milk (1.033-1.038) and the fat content (4-7%) are higher than those of cow's milk, a considerable quant. of the former may be added to ordinary skim milk, which has been watered, and the resulting mixt. still have the d. and % fat of normal milk. The d. at  $15^{\circ}$  of the serum obtained by Molinari's method, from sheep's milk, is between 1.026 and 1.029, which is about the same as for the serum from cow's milk; thus the adulteration by skimming. then adding water and sheep milk, can be readily detected in the serum. Tables showing the d., % fat, % solids and d. of serum of various proportions of milk, water and sheep milk are given.

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Pasteurization of Babies' Milk by Boiling at Low Pressure in the Hamburg Pasteurizer. R. Hanne. Gesundh. Ing., 34, 489.—Pasteurization of babies' milk in bottles by b. at 60° and at low pressure, insures absolutely satisfactory results. A lower temp. cannot be recommended because it does not guarantee a complete removal of bacteria. The lower the b. temp., the longer the b. will have to be extended. The pasteurization app. of Rud. A. Hartmann, Berlin, which has been used for these expts., makes uniform conditions possible. The forma ion of a fine film, which may serve as a protection to bacteria, is avoided by the continuous boiling up of the milk. Elimination of vegetative forms of bacteria, killing off the less resistant types of spore-bearers and sterilizing the neck and head of the bottle are assured. No biologic change could be detected in the milk. This pasteurization combines the advantages of the old pasteurization system with the one of sterilization without possessing certain disadvantages of either.

Arthur Lederer.

A Study of Milk Curd. R. WINDISCH. Kgl. Ungar. agr.-chem. Versuchst., Keszthely. Z. Nahr. Genussm., 21, 613.—Com. samples of this product (resembling our cottage cheese) were examined for moisture (123 samples, 63.45-75.78%, av. 70.05%), ash (95 samples, 0.48-1.05%, av. 0.698%) and fat (79 samples, 0.42-15.55%, 30 less than 5%, 34 between 5-10%, 1 more than 10%).

A. F. Seeker.

Verification of the Purity of Linseed Oil Cake. L. VUAFLART. Stat. agron. pas de Calais. Ann. jals., 4, 381-8.—The microscopic exam. of linseed cake for adulteration is described. A discussion is given of the significance of certain chem. detns. as N content, fat, cellulose in assisting in the interpretation of the microscopic findings. Mucilage seems to increase with the N content, but its detn. is inaccurate in such a product and is of no value in judging the purity of linseed cake. R. F. BACON.

Report on Fruit and Fruit Products. A. W. Blair. U. S. Dept. Agr., Bur. Chem., Bull. 137, 56.—This is a report of coöperative work on the detn. of moisture in blackberry jelly and fresh pineapple by the following methods: vacuum drying at 100° and 70°; provisional A. O. A. C. method for molasses (Bull. 107, revized, 38) using sand or Pb shot; Lowenstein alc. method (C. A., 3, 1971); vacuum method over H<sub>2</sub>SO<sub>4</sub>. Results by the last named method were entirely too low. It is believed that with fruits and fruit products the vacuum oven will probably give better result. than the other method.

P. B. Dunbar.

Studies on Fruit Respiration. H. C. GORE. U. S. Dept. Agr., Bur. Chem., Bull. 142.—The chief features of the bulletin are included in the following summary: (1) An absorption app. and a constant-temp. incubator suitable for use in the study of the respiration of fruits. (2) Measurements of the rate of respiration at different temps. of berries, peaches, apples, grapes, etc. (3) The respiratory intensity of these fruits varied greatly, but when the data were plotted similarly shaped curves were formed. (4) No correlation between comp. or size and respiratory activity appeared. In general, however, fruits which grow and mature quick y and soon become overripe respire rapidly. This is true of most of the small fruits. On the other hand, fruits having a long growing season and maturing slowly, as the citrus fruits, are very inactive physiologically. Peaches, plums, apples, pears, and grapes are intermediate in this respect. (5) When plotted as logarithms, the values expressing the intensity of respiration lie principally along straight lines; the curve therefore represents approx. the equation of the relation between respiratory activity and temp. The form of this equation is  $\log y = \log y_0 + at$ , in which the consts.  $y_0$  and a could be readily detd. by inspection. yo varied for each kind of fruit. a varied slightly from one fruit to another and was equal to  $0.0376 \pm 0.00044$ , the value 0.00044 being the probable error. (6) The rate of respiration increased on an average of 2.376 times for each 10° rise in temp. for 49 sets of detns. with 40 different kinds of fruits. (7) The general

equation expressing the effect of temp. on the respiration or fruits is  $\log y = \log y_0 + at$ , or  $y = y_0$  10°<sup>4</sup>, where y is the rate of evolution of CO<sub>2</sub> expressed as mg. per kg. of fruit per hr. at temp.  $t^\circ$ ;  $y_0$  is the respiration rate at 0°, and  $a = 0.0376 \pm 0.00044$ . (8). When CO<sub>2</sub>-free air was aspirated thru jars containing peaches attached to the tree and detached therefrom, resp., and the amts. of CO<sub>2</sub> given off by the fruit detd., no stimulus in the rate of respiration due, to picking, could be detected; if such change occurs it is but small. (9) Assuming that fruits follow the equation in (7), that the heat resulting from the respiration and the sp. heat of the fruit are known, and that the fruit is held under adiabatic conditions, an equation is derived from which the temp. of fruit t'' may be calc. after a given time T, starting at a known temp. t'. This equation is as follows:  $t'' = t' - [\log (1 - mT)/a]$  where m is the product of several constants  $(ky_i, a,$  and  $\log_2 10$ , in which k is the temp. rise accompanying the formation of 1 g. of CO<sub>2</sub> from sucrose (equiv. to 0.00285°),  $y_i$  is the respiratory activity of the fruit at t'°, and  $\log_2 10$  equals 2.3026.

W. D. Bioslow.

Report on Vegetables. John Phillips Street. U. S. Dept. Agr., Bur. Chem., Bull. 137, 122.—The relative with of peas and liquor were detd. in 111 samples and the results compared with the standards given by Bitting (C. A., 3, 1778). In 61% of the samples the % of peas was low. The appearance and characteristics of the various classes of peas are described in detail and the analyses of both peas and liquors are given. The average analysis of each grade of peas as shown by label is stated in tabular form. Aside from physical appearance, the low water content and high starch content of soaked peas, and to a less extent of peas canned in quite a mature state, are emphasized.

W. D. Bigelow.

Methods for the Analysis of Medicated Soft Drinks. H. C. FULLER. U. S. Dept. Agr., Bur. Chem., Bull. 137, 190-4.—Total solids and ash are detd. with a 25 cc. sample; alc. is detd. with a 100 cc. sample, any flavoring oils being removed from the distillate by means of petroleum ether and NaCl and a second distillation made; sugars are detd. by the official methods. Caffeine is detd. by digesting 50 cc. with NH4OH, shaking out 4 times with CHCl2, evapg., dissolving in dil. H2SO4, shaking out 5 times with CHCla, again evapg. the caffeine in acid soln. with I, filtering, decomposing the I compd. with H.SO, and shaking out the pure caffeine from NH.OH soln. with CHCl.. Cocaine is detd. by treating 200 cc. with NH,OH, shaking out with Prolius mixt., evapg. the solvent, dissolving the residue in dil. H<sub>2</sub>SO<sub>4</sub>, shaking out with CHCl<sub>2</sub> to remove the caffeine, then adding NH,OH and shaking out with petroleum ether, the residue left on evapn. is weighed and then tested for cocaine by the physiological, Et benzoate, and microchemical tests. In the case of an "extract" or "flavor" the product is first evapd., extracted with EtOH, the alc. soln. evapd., and the residue treated as above. Phosphoric acid is detd. by pptg. the ammoniacal soln. with magnesia mixt., dissolving the ppt. in dil. HNO2, pptg. with molybdate and finishing in the usual way. Provision is made for the detection of benzoic and salicylic acids, saccharin and dye-H. C. FULLER. stuffs.

Analysis of Liquids Containing a Large Proportion of Tartaric Acid and a Small Amount of Glycerol and Tannin. G. Hinard. Ann. jals., 4, 391.—It was necessary to analyze liquids containing about 100 g. tartaric acid, 10 g. glycerol and 3 g. tannin per 1. The large proportion of acid made certain modifications necessary particularly in detg. the ext. and glycerol. Evapn. on the water bath, followed by the drying oven at 100° did not suffice. The last traces of water are driven off from the tartaric acid with great difficulty. At 120–125° the acid is changed and gradually loses wt. Difficulty was also found using a vacuum. The method finally employed consisted in adding the liquid to calcined pumice stone in a small dish. This was then placed in a vacuum desiccator with H<sub>2</sub>SO<sub>4</sub> above and below. After 24 hrs. the H<sub>2</sub>SO<sub>4</sub> was replaced

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with  $P_2O_3$ . The desiccator was then heated to 50°. Accurate results were obtained with known mixtures. The glycerol was extracted as follows: After removing the acid as K bitartrate and filtering, freshly pptd. Pb(OH)<sub>3</sub> suspended in water was added. The water was expelled at 50° and the glycerol taken up with alc. Pb was pptd. by  $CO_3$ , and ethyl ether, and pure dry  $K_2CO_3$  added. The latter causes rapid clearing of the soln. After standing some time the liquid was filtered and evapd. at 50° and the residue placed in a vacuum at 50° with  $H_2SO_4$  or  $P_2O_3$  to const. wt. Good results. Tannin is detd. volumetrically using starch-I, after neutralizing the tartaric acid with NaHCO<sub>3</sub>.

The Fiehe Test as a Means of Judging the Purity of Honey. H. LUHRIG AND A. Scholz. Chem. Untersuch.-amt., Breslau. Z. Nahr. Genussm., 21, 721.—Upon heating a 66% sol. of sucrose with varying amts. of citric, tartaric, malic, formic, acetic. oxalic, lactic, butyric, propionic, salicylic, HCl and HF acids it was found that all of these are capable of causing a positive Fiehe test in the product. Abrastol acts similarly while valerianic and benzoic acids, acetaldehyde, propylaldehyde and hexamethylenetetramine produce negative results. A pure honey (sucrose 0.8%, Fiehe test negative) could not be made to produce a positive test by heating at 80° for 1/2 hr. with any of the above substances excepting H<sub>2</sub>C<sub>2</sub>O<sub>4</sub>, HCl and HF. The same results were obtained after adding 3% of sucrose to the honey. After adding 7% of sucrose a positive test could be obtained by treatment with the following: H,C,H,O, (0.9%),  $H_2C_2O_4$  (0.2%),  $H_4C_4H_4O_6$  (1.4%),  $H_2C_4H_4O_8$  (2%), abrastol (1.8%). A 66% sucrose sol. gave a positive test after standing for 12 days at room temp, with 0.3% HCl (3 days with 0.4% HCl). HF (0.5%) required 6 days at room temp., and H<sub>2</sub>C<sub>2</sub>O<sub>4</sub> (5%) 10 days. In view of the above facts and considering our ignorance of the nature of the acids naturally present in honey, and of the treatment received by the product before it reaches us, the author considers it inadmissible to assert that a sample is adulterated on the ground of a positive Fiehe test. The latter serves merely to render the product suspicious and necessitates a thoro exam. Analyses of 91 samples of honey are given. A. F. SEEKER.

Honey and its Analysis. F. MUTTELET. Ann. chim. anal., 16, 299-305; see C. A., 5, 1131 and 2275.

J. J. M.

The Application of the Quantitative Precipitin Reaction in Honey Investigations. J. THÖNI. Mitt. Lebens. Hyg., 2, 80-123; thru Chem. Zentr., 1911, I, 1888.—Von Reigler (Oesterr Chem.-Ztg., 1902, 97) succeeded in obtaining an anti-serum which caused pptn. Langer (C. A., 4, 332; and Galli-Valerio and Barnard, C. A., 4, 3098) did much work on this subject. The author confirmed the findings of Langer (C. A., 4, 332). As factors which det. the formation of a ppt. there are principally the degree of conc. and the amt. of honey soln., as well as the potency and amt. of the antiserum of bees honey. The testing of 90 honeys and sugars gave: 1. No ppt. was formed in the sugars. 2. That artificial honeys, like sugars, gave either no ppt. or only small amts. (in 10-15% solns. traces up to 2 mm. high). 3. That in genuin bee honeys, using the same anti-serum of bees honey, the height of the layer of ppt, varied but little, and a pronounced ppt. was always formed in a 1% soln. The total amt. of the ppt., using the same serum, was in different series of expts. as follows (expressed in mm.): 13 tests, max. 59.0, min. 43.0; 3 tests, 40.5 and 33.5; 8 tests, 17.5 and 10.75; 2 tests, 13.0 and 10.5, resp. 4. In mixts. of genuin and artificial honey the height of the ppt. decreased with the decrease of true honey. 5. That "sugared" honeys gave distinctly less ppt. than genuin pure bees honey. 6. In fermenting honeys the amt. of the ppt. did not decrease as compared to that with true honey. The paper closes with exact directions for the use of seriological method. The method is in many ways superior to chem. and phys. methods. R. F. BACON.

Food Inspection Decisions. Published separately by the United States Department of Agriculture. F. I. D. 134. The Labeling of New Orleans Molasses: Product is restricted to molasses produced in La. and may bear a further appropriate statement descriptive of quality or grade such as "open kettle," "first centrifugal," "second centrifugal," "black strap," etc. F. I. D., 136. Labeling of Chocolate and Cocoa: Chocolate, plain, bitter, liquor or coatings contains not more than 3% ash insol. in water, 3.05% crude fiber, 9% starch, and not less than 45% cocoa fat. Sweet chocolate conforms to the same specifications with the addition of sugar and with or without added cocoa butter, spices and other flavors. Cocoa is plain chocolate deprived of a portion of its fat and pulverized and the standards of comp. are the same after correction for fat removed. Sweet cocoa is cocoa containing not more than 60% added sucrose and the standards of comp. of plain chocolate apply, taking into consideration the % of added sugar and of fat removed. Milk chocolate and milk cocoa contain not less than 12% of milk solids. Nut chocolate must contain substantial quantities of nuts. Milk and nut chocolate containing added sugar should be labeled "sweet milk chocolate," etc. Cocoa treated with alkali or alk. salts so as to increase mineral content should be labeled as containing added mineral ingredients, stating the amt. Cocoas and chocolates containing free alkalies are adulterated. Cocoas treated with alkali should not be designated as "soluble." F. I. D., 137. The Use of Charlock as a Substitute for Mustard: A mixture of mustard and charlock must be so labeled. F. I. D., 138. Saccharin in Food: After Jan. 1, 1912, the presence of saccharin in foods is not permitted. W. D. BIGELOW.

Report on Sugar and Molasses. H. P. AGER AND R. S. HILTNER. U. S. Dept. Agr., Bur. Chem., Bull. 137, 160.—The report covers the cooperation work for the A. O. A. C. during 1910. Four methods of estg. water were tried, including drying in vacuum oven at 70°, drying in an oven at 100° for 10 hours, by Brix spindle and by refractometer. Polarizations were obtained using wet and dry lead subacetate; and Herle's soln. both in necessary amts. and in excess. Com. glucose was estd. by the regular official method. The referees recommended a further study of the methods tried.

A. H. BRYAN.

Effect of Formic Acid on Health. Pure Products, 7, 438-42.—The Scientific Committee for Medicin under the Prussian Government has found that HCO<sub>2</sub>H is absorbed without any deleterious action on the organs of the body except corrosion when in sufficient conc. It is exactly analogous to HOAc except that it is more corrosive. The committee disapproves, however, of the general use of HCO<sub>2</sub>H as a preservative.

W. D. Bigelow.

Report on Preservatives. Quantitative Estimation of Sodium Benzoate. P. B. Dunbar. U. S. Dept. Agr., Bur. Chem., Bull. 137, 108.—This is a report of cooperative work on the detn. of BzONa in jellies, jams and salt codfish by a slight modification of the method described in C. A., 4, 2338. In the case of jellies and jams, the material is made alk. with milk of lime instead of NaOH. Codfish is made alk. with NaOH and the BzONa extracted with a definit vol. of water. An aliquot of the soln. so obtained is satd. with NaCl, filtered, acidified, extracted with CHCl, the soln. evapd. and the residue of BzOH titrated. The recoveries reported by the collaborators are in most cases very close to theoretical. Mohler's test for BzOH as modified by von der Heide and Jakob (C. A., 4, 1523) was studied and found to give a very clear test with as low as 0.5 mg. BzOH.

Color of Flour and a Method for the Determination of the "Gasoline Color Value."

A. L. WINTON U. S. Dept. Agr., Bur. Chem., Bull. 137, 144.—The color of flour is due (1) to mechanical impurities (2) to "oil sol. color." Flour extracted with Et<sub>2</sub>O, became dead white, and resembled starch. It lacked coherence and flavor and could

not be slicked for pekarizing. Bleaching reduces the wholesomeness of flour and injures its flavor. No direct method is available to det. the color due to mechanical impurities, altho these are in general proportional to the ash, fiber, fat, and protein, the ash being generally dependent upon to det. the milling grade. The author dets. the oil sol. color as follows: Place 20 g. flour in a wide mouthed 120 cc. bottle, add 100 cc. colorless gasoline, stopper, shake 5 min. Let stand 16 hrs., filter, and compare the color of the gasoline in a Shreiner colorimeter with a 0.005% soln. of K<sub>2</sub>CrO<sub>4</sub> this standard soln. having the gasoline color No. 1. The amt. of color varies with the variety of the wheat, Nebraska hard wheat having about twice as much color as that of Mich. or Missouri soft winter wheats, while Minn, hard spring wheat is intermediate. The patents and clears from the same milling do not differ appreciably from each other in gasoline color value. Aging lowers the color value. In the winter months this became apparent after 10 weeks. During the summer it was much more rapid. After 30 weeks three-fourths of the color had disappeared. Bleaching with nitrogen peroxide gave the same whitening at once that was obtained by aging for 30 weeks. If the history of the flour is known, the gasoline color number is of value in detecting bleaching. R. F. BACON.

Thermal Sulfurous Stations. P. CARLES. Ann. fals., 4, 388. R. F. B.

Report on the Separation of Vegetable Proteins. R. HARCOURT. U. S. Dept. Agr., Bur. Chem., Bull. 137, 149.—Work was done on 2 greatly differing samples of flour, to det. whether the prescribed methods of sepg. the proteins gave any indication of the quality of the flour. Total N, salt sol. N, and alc. sol. N were detd. The results of different workers are in good accord, but apparently have no connection with the quality of the flour.

D. S. Prata

The Milling Value of Water-soaked Wheat. GRO. A. OLSON. Am. Food J., Apr. 15, 1911.—Wheat was soaked in H.O and allowed to germinate. In 1 lot the acrospire was allowed to grow 1/4 the length of the kernel, in 1 lot to an equal length, and in 1 lot to twice the length. The lots were then dried, weighed, and ground into flour, bran, and shorts. The decreased yield of wheat due to germination was from 0.55-5.0%. The loss in milling products was from 1.53-8.92%. The diastatic power of the germinated wheat flour as compared to ordinary flour was increased from 3-20 times. The gluten decreased from 14.19-2.06; 13.40-0.03; 10.44-0.28; 13.43-0.38 in different wheats. A study of the effects of germination on other nitrogenous constituents of the wheat was made. Alcohol sol, and amide N increased with germination, albumin and globulin were fairly constant, glutenin showed a marked decrease. Baking tests were made. Loaves prepared from germinated wheat were sticky to the touch, sweet and were very porous and collapsible. The addition o. a small amt. of flour (up to 10%) from germinated wheat to ordinary has a beneficial effect on the baking qualities of the flour, giving loaves of increased size and H<sub>2</sub>O content.

R. F. BACON.

Catchup. S. D. State Food and Drug Dept., Bull. 21.—Dealers are warned against the sale of catchup made from spoiled material.

W. D. BIGELOW.

Concrete Cisterns for Vinegar. H. B. BONEBRIGHT. Colo. Agr. Expt. Sta. Eng. Record, 64, 103.—Expts. on various lining materials are described. Cement tile proved unsatisfactory. Cement cisterns proved satisfactory when washed with 2 coats of neat cement grout, and treated with paraffin rubbed in hot. The coating must not be injured.

LANGDON PEARSE.

Report on Tea and Coffee. M. E. JAFFA. U. S. Dept. Agr., Bur. Chem., Bull 137, 105.—Several modifications of the Hilger and Fricke and the Spencer methods for the detn. of caffeine were compared. The results obtained by weighing the residue

were uniformly too high and more favorable results were secured by calculation from the detn. of N. W. D. Bigglow.

Occurrence and Estimation of Tin in Food Products. BERNARD H. SMITH AND GEORGE M. BARTLETT. U. S. Dept. Agr., Bur. Chem., Bull. 137, 157.—Weigh 50 g. of fish or 100 g. vegetables into a porcelain dish and dry over night. To 75–100 cc. conc. H<sub>2</sub>SO<sub>4</sub> heated to fuming in a Kjeldahl flask add small portions of the sample till frothing ceases. After cooling add gradually 25 cc. HNO<sub>2</sub> (d. 1.42). Red fumes are given off and the flask becomes warm. Allow to cool again, add 25 cc. HNO<sub>2</sub> and heat gently till all HNO<sub>2</sub> fumes are expelled. Boil this soln. which now should be perfectly homogenous, add 10–15 g. K<sub>2</sub>SO<sub>4</sub> and boil till soln. is colorless. Transfer to 800 cc. beaker, dil. to 600 cc. and bring to a boil. Filter off the hydrated SnO<sub>2</sub>, return to flask, add 20 cc. satd. NaOH and an equal vol. of water and boil several mins. Wash the Na stannate thus formed into a beaker, acidi y with HCl and ppt. with H<sub>2</sub>S as directed in Bull. 107, Rev., p. 69.

C. W. Clark.

Report on Water in Foods. P. F. TROWBRIDGE. U. S. Dept. Agr., Bur. Chem., Bull. 137, 138-40.—The work consisted of a comparison of the official method for the detn. of water and the vacuum method without heat. The latter method consists in drying to const. wt. in a vacuum desiccator over H<sub>2</sub>SO<sub>4</sub>. Three samples of ice cream required 11, 5, and 10 dryings respectively to get them to const. wt. by the official method. Two dryings were sufficient for each sample by the vacuum method. The latter method gave better duplicates and higher results. Similar results were obtained on 12 samples of corn meal. The av. increase in moisture by the vacuum method over the official method was 1.210%. The vacuum method also has the advantage of leaving the sample in better condition for the subsequent detn. of the ether ext.

C. P. WILSON.

The Fermenting of Fodders and the Consequent Loss in Food Value. F. TANGL AND S. WEISER. Landw. Vers. Sta., 74, 263-342.—On July 15, 30,000 kg. of fresh alfalfa containing 41% of H<sub>2</sub>O were placed in an open pit and on March 15, 20,010 kg. containing 48% H.O were recovered. The non-alb. N material showed an increase of 33%, while all foodstuffs showed a diminution. The loss in storing was 75% food material and 25% non-food material, 75% of the loss in organic matter was carbohydrates. Of the consumable food material there was lost; dry material 52.4%, raw protein 53.7%, pure protein 75.6%, crude fat 59.9%, crude fiber 56.5%, N-free ext. 51.1%. Sugar beets stored in rick from the end of Oct. to the end of June showed a diminution from 6556 kg. dry material to 5822 kg. The loss was entirely organic, and was mostly consumable material as follows: Organic 56.8%, pure protein 54.8%, crude fat 100%, crude fiber 40.2%, N-free ext. 57.1%, starch value 50.8%, and the content of oxalic acid fell from 2.02-1.04%. The non-consumable feeding material had increased. Maize straw in silo only changed in feeding value to an insignificant E. C. LATHROP. extent.

The Suppression of Fraud in Precious Stones, Furs and Merchandise of every Character. Anon. Ann. fals., 4, 353-5.—The French law of April 1, 1905, is general and provides against deception concerning the nature, comp., quality, or origin of all classes of merchandise.

R. F. BACON.

A Modification of the Method for Crude Fiber. MORGAN O. SWEENEY. U. S. Dept. Agr., Bur. Chem., Bull. 137, 157.—Place 2 g. sample in a wide mouth 1. Erlenmeyer flask, inserting a small air condenser to prevent conc. due to loss of steam. Add 200 cc. 1.25% soln. of boiling H<sub>2</sub>SO<sub>4</sub> and boil for 30 min. Neutralize with a 10% soln. of NaOH, using phenolph. as indicator. Add at once 200 cc. 2.65% soln. of boiling NaOH and boil for 30 min. Filter rapidly thru a linen cloth and wash with

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boiling water. Transfer the fiber to a tared Pt Gooch crucible and wash with alc. and ether. Dry at 100° and weigh. Ignite the dried residue and weigh again, the loss in weight giving crude fiber. Tables are given showing that duplicates by this method agree more closely than by the official method and results by this method are very near those by the official method. The time required for this detn. is shorter than for the old method and the most difficult and disagreeable part of the present method is eliminated.

C. W. Clark.

Action of Acids and Salts on Gelatin (PROCTER). 29.

Analysis of Copra. 27.

A New Adulterant (Kraemer). 17.

Apparatus for Fat Extractions (Volrath). 1.

Coconut Oil in Edible Fats (Arnold). 27.

Coconut Oil (Richardson). 27.

Constituents of Milk (Tangl, Zaitscher). 13.

Cottonseed Cake (Golding). 27.

Determining Ether Extract (Leach, Hiltner). 7.

Determining Fat in Cotton Products (MacNider). 27.

Examination of Oils (Duperthuis). 27.

Inorganic Plant Constituents (Shedd). 11.

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GREENISH, H. G.: Microscopical Examination of Foods and Drugs. Blakiston. 386 pp., \$3.00.

Guillin, R.: Analyses alimentaires. 480 pp., 6 Fr.

TEICHERT, K.: Die Analyse der Milch und Milcherzeugnisse. 2 Aufl. Berlin: J. Springer. 8°, 81 pp., 2.40 M.

Brit., 15,313, June 25, 1910. J. HUTCHEON, 18 St. Mary St., Edinburgh. Food-stuffs consisting of a cooked mixt. of oat-meal flour, pea-meal flour, a salt or salts, and if desired any other flavoring agent, margarin, or butter and milk sugar, the whole admixed with lactic ferment powder. Further details are specified.

Brit., 23,293, Oct. 8, 1910. H. D. GILLBANKS, Victoria Bldgs., Bury, Lancashire. A food preparation of high nutritive value, consisting of an intimate mixt. of meat extract and milk, powder obtained when meat ext. is allowed to act on milk powder, the latter being largely in excess of the former, with or without the addition of a peptonizer and with or without the addition of other nutritive substances.

Brit., 4,308, Feb. 20, 1911. R. Moll., Beim Strohhause 31 E, Hamburg 5, Ger. Preserving marxipan and similar confection by pressing the substance free from bubbles into a glass vessel level with the edge thereof, covered with an acid-resisting layer of paper or paste-board and hermetically closed by a closely fitting cover and thereupon heated to 120° for 10-30 mins. in a steam or H<sub>2</sub>O bath.

Brit., 6,160, Mar. 11, 1911. F. KOSTER, 18 Graeme Road, Enfield, Middlesex. In the treatment of separated milk to produce a solid material therefrom, the milk, with or without coloring matter, is first warmed then curdled with rennet, the curds resulting from such treatment being washed, compacted, comminuted, subjected to pressure, and hardened.

## 13. NUTRITION.

## C. F. LANGWORTHY.

Digestion and Resorption of Nucleic Acid in the Gastrointestinal Tract. E. S. London, A. Schittenhelm and Karl Wiener. Path. Lab. Inst. Exp. Med., St. Petersburg and Lab. of Clinic Med., Erlangen. Z. physiol. Chem., 72, 459-62.—Expts. were performed with ileum fistula. On feeding thymus nucleic acid, guanylic acid could be sepd. from the intestinal juice. Guanosine was also sepd. and identified, which lends proof of the existence of nucleosides in thymus nucleic acid similar to those sepd. from other nucleic acids by Levene and Jacob. G. M. Meyer.

Influence of Colloidal Sulfur on the Sulfur Exchanges of the Organism. Contribution to the Mechanism of Sulfoconjugation. L. C. MAILLARD. Compt. rend. soc. biol., 70, 940-3.—Colloidal S administered per os to rabbits is rapidly absorbed and eliminated in the urin as neutral S inside of 24 hrs. Following this elimination ethereal S rose to triple the normal value.

I. K. Phelps.

Decalcifying Action of Oxalic Intoxication. SARVONAT AND CH. ROUBIER. Compt. rend. soc. biol., 71, 104-5.—Guinea pigs, treated subcutaneously with small doses of oxalic acid until they died, were found on analysis of the skeleton and soft parts, separately, to have the Ca content of each markedly reduced.

I. K. PHELPS.

Frozen Meat. GILBERT ANDERSON. J. N. Zeal. Dept. Agr., 2, 278-9.—The real nutritive matter in fresh and frozen meat is almost identical. G. A. ROUSH.

Further Investigations on the Influence of Stimulants on Milk Secretion. G. FINGERLING. Landw. Vers. Sta., 74, 163-82.—Molasses increased milk secretion by goats but no more than other feed rich in stimulating material. Feeding mixtures with pleasant taste had no effect on the amt. or quality of the milk.

M. X. SULLIVAN.

The Influence of Different Watery Feeding Material on the Amount and the Constituents of Milk. F. TANGL AND A. ZAITSCHEK. Landw. Vers. Sta., 74, 183-249; Chem. Zentr., 1911, I, 1077.—The belief that watery feeds make a thinner milk than dry feed was not confirmed. With wet and dry feeds there was no difference in the yield of milk nor in the constituents, altho the av. water consumption of the cows in the water period was 30% higher than in the dry period. Using cows the action of single feeds is as follows: (1) Fermented maize residue. The acid content of the milk was not increased but the fat content of the milk was raised. (2) Pumpkins: The amt. of milk was diminished. The dry substance of the milk did not change, for, while the ash, protein, and fat content was increased, the sugar content was lowered. The milk was slightly yellow. (3) Turnips: The turnips increased the yield of milk and of protein. The milk had no turnip taste. (4) Potato: The amt. of milk was lessened but the constituents were unchanged. The N content of the milk did not decrease in spite of the fact that the cows in the potato feeding period received much less digestible crude protein and only 1/2 as much pure protein. (5) Alfalfa: The amt. of milk was considerably increased. The N content was increased while the fat was lowered somewhat. In the lactation period the amt. of milk diminished while the % of N containing substance and fat increased; the milk sugar and ash remained almost unchanged. Of the day's milk 55% came from the morning milk and 45% from the evening. The latter contained more fat than the former. M. X. S.

The Influence of Feeding Materials on the Constitution of Milk Fat. A. ZAITSCHEK. Landw. Vers. Sta., 74, 250-62; cf. preceding abstr.—The sap. no., the R.-M. no., I no., n, the m. p. and the color of the butter fat were observed. With the turnip diet the sapon. no., and R.-M. no. were higher and the I no. and n lower than with the dry

feed. The potatoes acted as did the turnips except that they raised instead of lowered the m. p. The alfalfa feed gave a butter fat of normal constitution like that obtained by dry feeding. Towards the end of lactation the sapon. no. and R.-M. no. of the butter fat fell while the I no., n and m. p. rose. After a change of feed the R.-M. no., I no. and n took their original values.

E. C. LATHROP.

# 14. WATER, SEWAGE AND SANITATION.

#### EDWARD BARTOW.

Report on Water. J. K. HAYWOOD AND W. W. SKINNER. U. S. Dept. Agri., Bur. Chem., Bull. 137, 42-5.—Report of cooperative work on analysis of an artificial mineral water by the methods given in Bur. Chem., Circ. 52. The results were so variable that the referee recommended further cooperative work with the same methods.

W. D. COLLINS

Hypochlorite of Lime Treatment Plant at Erie, Pa. C. A. JENNINGS. Eng. Record, 64, 100.—Lake Erie water is normally low in turbidity, organic matter and bacterial content. A "hypo" treatment plant, installed during the typhoid epidemic in 1911, consists of 3 reinforced concrete tanks, orifice boxes, and grids in the intake pipes. The stock soln. contains 0.4% available Cl. Only clarified soln. is used. 7-10 lbs. Ca(OCl), are used per mil. gals. water, or 0.32 pts. per mil. of available Cl. No sample of treated water has shown B. coli. No complaint of taste or odor has been received. LANGDON PEARSE.

Water Supply and Sewerage of the National Rifle Range, Camp Perry, Ohio. R. W. Pratt. Eng. News, 65, 758-60.—Sewage passes to a collecting well and is pumped from here to 4 sand filters. There is no preliminary treatment. Since the camp is mostly in use during the summer, when bacteriological action is best, the area provided is in the proportion of 8000 persons to the acre of filtered surface, instead of 1000. The filter effluent is treated with chloride of lime. The water purification plant consists of 2 settling basins, holding about 2 hours flow; 2 filters, each of an area of 1000 sq. ft.; a control chamber and clear water basin. The sand removed from the filters is not washed, but is replaced with new sand from the beach. The bacterial reduction has been interfered with by the short settling period not adequately removing the suspended matter. The average rate of filtration is 4 mil. gal. per acre daily.

EDWARD BARTOW.

Operating Results at the St. Louis Water Coagulating Works. Eng. Record, 64, 162.—The variations in the chem. constituents and bacterial contents are given. The average total cost, per mil. gals. was \$4.775. The coagulant cost was \$1.928 for 2.70 g. per gal. Fe sulfate, and \$1.875 for 5.77 g. per gal. lime. The past year has been distinguished by low average turbidity and an increase in the dissolved solids, as well as the alkalinity. Typhoid statistics are given.

LANGDON PEARSE.

The Potash Works in the Elbe and Weser Districts and the Water Supply of Magdeburg and Bremen. P. KRISCHE. Gross-Lichterfelde. Chem. Ind., 34, 326-8.—A consideration of statistics gathered from 1880 to the present time showing that the rapid growth of this industry in the above districts and the consequent increased disposal of waste by-products in the rivers has had no detrimental effect on the water supply of the above cities for use in the home.

E. J. Crane.

The Bacterial Control of Sewage Purification. LEDERER AND F. BACHMANN. Eng. Record, 64, 89-90; Ill. Water Supply Assu., 1911.—The danger is emphasized of relying on bacterial control of sewage purification.

LANGDON PEARSE.

Distillation of Dried Sewage Sludge and the Manufacture of Illuminating Gas.

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A. BAYER AND L. FABRE. Rev. gen. chim., 14, 199-205.—Seventy-five analyses of dried sludge made at Brunn, Austria, give as an average the following:

Per cent. C. H. N. O. Ash. Phosp, acid. K. Heat units. Mean........ 31.04 3.95 2.87 20.73 41.41 1.63 0.42 2908

Distillation test of 55 kg. of dried sludge in 2<sup>1</sup>/<sub>3</sub> hrs. of complete distillation produced 23.8 cu. m. of gas per 100 kg. Comparative analyses of sludge and oil gas are:

Sludge gas.	CO <sub>3</sub> .	hydro- carbons.	Methane.	CO.	н.	О.	N.	Heat units.
1907	2.2	4.8	18.1	17.5	44.2	0.8	12.0	4122
1908	2.2	7 · 3	23.2	14.4	41.7	0.8	7.9	4759
Oil gas, Brunn	2.2	3.1	26.8	II.I	50.0	0.6	6.2	4950

The conclusions and recommendations were: (1) The sludge will be extracted by a continuous mechanical system, producing from the wet sludge a dry sludge containing 25% dry matter in flakes; (2) the drying of wet sludge takes 2 kg. oil per 100 kg. wet sludge; (3) distillation of sludge yields 23 cu. m. of good illuminating gas with 0.75 kg. NH<sub>2</sub> at a minimum per 100 kg. of dry sludge; (4) the use of dry sludge to produce illuminating gas and NH<sub>2</sub> is the best way to recover fresh material from sewage; (5) the clarified water will be clear and easily treated biologically, on sprinkling filters.

LANGDON PEARSE.

Sewage Distribution or Sprinkling Filters. W. H. DITTOR. Munic. J. Eng., 31, 73-9.—A detailed account of tests made to det. the dosing and distribution for the sprinkling filters at College Hill, O. (cf. C. A., 5, 1479). An equalizer with inclined sides was finally adopted to insure even distribution.

LANGDON PEARSE.

East San Jose Sewage Disposal Plant. Anon. Eng. Record, 64, 140. L. P.

Milwaukee Refuse Destructor. Anon. Munic. J. Eng., 31, 137-40; Eng. Record, 64, 186-7.—A general description of the method of operation, the data obtained on test runs, the steam production, with a synopsis of the recommendations of the Efficiency Bureau. The actual costs of operation are summarized (\$2.1683 per ton of garbage burned) (cf. C. A., 5, 1814).

LANGDON PEARSE.

Harbor Water for Flushing New York Streets. Anon. Eng. Record, 64, 142.—
Portable pumping plants have been built on 4-wheeled carts to pump 200 gal. per min., under 50 lbs. pressure per sq. in. The water is drawn direct from the harbor. One case of injury to asphalt has resulted, where brine from warehouses has increased the amount of NaCl.

LANGDON PEARSE.

Inhibition of Growth by Certain Organic Substances in Solution and Vapor. H. STADLER. Arch. Hyg., 73, 195-217.— Expts. with Staphylococcus aureus, B. coli and pyocyaneus show that growth inhibition increases with mol. wt. in the aliphatic alcs. Lower homologs of aliphatic aldehydes show lessened inhibition with increased mol. wt. Substitution of O by S increases inhibitory action. Solns. and vapor of organic compds. have the same inhibitory power if they have the same partial pressure, except in case of chem. combination between the inhibitory substance and the culture medium. A table is given of concs. necessary for complete inhibition.

G. R. HENRY.

Researches on Tuberculosis of Milch Cows. GIUSEPPE COSCO. Arch. farm. sper., 12, 90-6.—An enumeration of the avenues of infection.

A. W. Dox.

Composition of Sea Water (CLARKE, STEIGER). 8. Determination of Nitrate Nitrogen (QUARTAROLI). 7. Pasteurization of Milk (HANNE). 12.

Simple Method for Nitrate and Nitrites in Water (TILLMANS, SUTTHOFF). 7. Utilization of Sewer Water (MUNTZ, LAINE). 15.

GEHRCKE, E., AND REICHENHEIM, O.: Das Dopplespektrum der Wasserstoffkanalstrahlen. Braunschweig: F. Vieweg & Sohn.

# 15. SOILS AND FERTILIZERS.

## F. P. VEITCH:

The Weathering of Silicates. H. STREMME. Landw. Jahrb., 40, 325-38.

M. X. S.

Humus. J. Hudig. van Bemmelen Gedenkboek, 1910, 308-21; Chem. Zentr., 1911, I, 1002.—In the peat settlements of Groningen and Drent, oat sicknesss has lately occurred. The leaves of plants, especially oats, became yellow in the sixth to eighth week of growth and the plant died. The cause of the sickness is to be found in definit organic constituents of the soil. The sick soils contained more lime and less SiO<sub>2</sub> than the normal soil.

M. X. Sullivan.

The Chemical Nature of Organic Nitrogen in the Soil. S. I. Joddi. J. Am. Chem. Soc., 33, 1226-41.—The total N extracted by b. HCl was on the av. 75.99%. The acid-sol. N consists of: Ammoniacal N 1.22-1.97%; nitric N 0.07-1.19%; N of acid amides 25.46-33.03%; N of diamino acids 12.21-13%; N of monoamino acids 22.20-55.66%. Boiling H<sub>2</sub>O extracted only 2.92 to 7.88% of total soil N. The acid amides and amino acids, which form the bulk of the HCl-sol. N are present in soil in some kind of combination. In case of soils containing comparatively fresh organic material the org. N (extracted) consists mainly of acids, amides and amino acids; in soils containing old organic material 47-60% of the compds. belong to other classes. The subject is treated in more detail in Res. Bull. 1 (1911), Iowa Sta. Coll. Agr. Exp. Sta.

M. X. S.

Report on Soils. J. G. LIPMAN. U. S. Dept. Agri., Bur. Chem., Bull. 137, 25.—

The report contains the results of the coöperative work on the detns. of total K and acidity in soils. The work on the detn. of total K consisted of a comparison of the Smith and the cobalti-nitrite methods. The results of the 2 methods compare very well and the referee considers the cobalti-nitrite method acceptable as a provisional method for the detn. of total K in soils. The work on soil acidity consisted of a comparison of the "Veitch" and "Suchting" methods. The results are variable and contradictory and strongly indicate the need of further work.

J. S. ROGERS.

The Alleged Refutation of the Lime Factor Theory. O. LORW. Landw. Jahrb., 39, 1005–9.—The reliability of Meyer's conclusion (see C. A., 5, 1149) is questioned on the ground that the plants were harvested at the blooming stage rather than maturity and also because too many plants were grown in each pot to secure normal development, and so do not parallel Loew's expts.

M. X. SULLIVAN.

The Dependence of the Maximal Yield on a Definit Ratio of Lime to Magnesia in Soil. D. MEYER. Landw. Jahrb., 40, 339-40.—An answer to Loew (preceding abstr.). The author holds that his own conclusions (C. A., 5, 1149) are correct.

M. X. Sullivan.

The Mechanical Analysis of Soil. W. E. Pratt. Bur. Sci., Manilla, P. I. Philipp. J. Sci., 6, 35-41.—The method of analysis is practically that outlined by the Bur. of Soils, U. S. Dept. Agr., Bull. 24. Some modifications of the method and the analytical results are given.

M. X. SULLIVAN.

Can Germany's Potash Monopoly be Broken? C. L. BREGER. Mining World, 34, 543-6.—There is a K area in the U. S. in Sevier Valley, near Richfield, Utah, in the form of the mineral alunite, K<sub>2</sub>SO<sub>4</sub>·3Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, and deposits in Chile. There is no saline area, however, in America which can furnish soluble K capable of competing with Germany. A possibility exists in ground feldspar. M. X. SULLIVAN.

Field Trials with Nitrogenous Manures from the Atmosphere. James Hendrick. J. Soc. Chem. Ind., 30, 522-4.—Field and pot expts., from 1905 to 1910, with Ca cyanamide and Ca(NO<sub>2</sub>)<sub>2</sub>, show that both fertilizers are comparable on grain crops with NaNO<sub>2</sub> and (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>. Ca(NO<sub>2</sub>)<sub>2</sub> gave each year better results than any of the other fertilizers. In pot expts. cyanamide retarded germination and was unsuitable in the field as a top dressing to growing plants since it spots and injures the foliage. Ca(NO<sub>2</sub>)<sub>2</sub> is especially suited for top dressing since it soon liquefies and finds its way into the soil. Its hygroscopic nature, however, is of great disadvantage in packing and handling it. Mixed with superphosphates or any other sol. phosphates it causes them to revert to an insol. state, a property which interferes with its use in mixed fertilizer.

M. X. Sullivan.

The Action of Different Proportions of Lime and Magnesia in a Soil on Higher Plants and Microorganisms. O. Lemmermann, A. Einecke and H. Fischer. Vers. Sta., Berlin. Landw. Jahrb., 40, 173-254.—(1) Expts. with 8 plants on 6 soils showed that as far as the yield was concerned it made little difference if the ratio of CaO to MgO varied greatly. (2) Even on soils which contain only 0.08% CaO and 0.04% MgO, 0.67% CaO and 0.09% MgO, resp., addition of Ca and Mg had little influence on yield of many plants. (3) The favorable action that lime often has on mustard is due as much to other factors as directly to the lime. (4) Grains are much poorer in CaO than the straw. There is less difference in the MgO content. The grain contains more MgO than CaO while the straw contains more CaO. (5) The constitution of the straw is more influenced by the CaO and MgO in the soil than is that of the grain. (6) With like yield the content of Ca, Mg, P,Oa in the plant may vary greatly. (7) The amount of Ca and Mg taken from fertilizer and soil is small. (8) With increasing Mg in the fertilizer and decreasing Ca, the Ca of the group decreases while P<sub>2</sub>O<sub>4</sub> and Mg increases. (9) The expts. do not show that a definit relation of Ca to Mg is especially favorable for plants or for microorganisms. M. X. SULLIVAN.

The Lime Requirements of Soil as Determined by Soil Investigations and Vegetation Experiments. O. Lemmermann, O. Foerster and A. Einecke. Vers. Sta., Landw. Jahrb., 40, 255-324.—The results and conclusions of Lemmermann and Fischer are corroborated (see preceding abstract). Other conclusions are: (1) In almost all soils vetch did poorly after vetch, while mustard after mustard gave better yields in the second year. (2) An unfavorable action of gypsum on legumes was not noticed. (3) Plants behave differently on the different soils towards lime fertilizer and acids in the soil. (4) The use of CaO by plants stands in no constant relation to the amt. taken from soil by extraction methods. (5) The taking of nutrient by the plant is self-regulated. Therefore a plant will not behave the same way on all soils. (6) Since it seems that material taken from the soil by the plant is returned to the soil again, the relation between the content of the plant and the results of soil analysis is not close. (7) The use of CaO by rye and vetch stands in a constant relation in one soil but varies in other soils. (8) The detn. of the CaO content of soil by the different methods gives no sure judgment of the CaO requirement. The detn. of the acidity of the soil is of greater importance. (9) There are more acid mineral soils than has been supposed. More attention should be given to the acid content of soils. M. X. SULLIVAN.

The Action of Phonolith Meal. W. KRÜGER, H. ROEMER AND G. WIMMER. Mitt.

Herz. Anhalt. Vers. Sta. No. 48; Chem. Zentr., 1911, I, 1076.—Phonolith meal and corresponding amts. of K salt were compared in pot expts. with summer rye, oats, rye grass, potatoes, and sugar beets and in field expts. with summer wheat and potatoes. In the field expts. a soil lacking in K was used. The phonolith meal had a certain K action which was lowest in the case of potatoes and rye, highest in case of oats and sugar beets, but always of much less effect than the K salts. On account of its cost, phonolith meal cannot be a substitute for K salts. For the pot expts. the phonolith meal used contained 10.22% total K and 3.05% K sol. in HCl and traces of water sol. K. That used in the field contained 17.12% total K, 3.01% sol. in HCl, and 0.06% sol. in water. The field expts. were repeated with similar results with phonolith meal containing only 7.73% K.

M. X. Sullivan.

Agricultural Utilization of Sewer Water. A. Müntz and E. Laing. Compt. rend., 152, 1814-8.—In tests of fields where sewer water is used the soil was enriched in N, P<sub>2</sub>O<sub>3</sub>, K, Ca, and Mg. To make sewer H<sub>2</sub>O a well balanced fertilizer it is necessary to add phosphates.

M. X. Sullivan.

Celery Sickness and Experiments for Combatting it. H. KLEBAHN. Mitt. deut. Landw., 1911, 6; Chem. Zentr., 1911, I, 1151.—There are 2 types of sickness, the spotted leaf and scabbing of the stems. Dip the seeds in 2% CuSO4. Disinfect the beds with dil. formalin or with phenostal. The disinfection of the field was too expensive.

M. X. SULLIVAN.

The Adherence of Insecticide Sprays of Lead Arsenate. H. ASTRUC, A. COUVERGNE AND J. MAHOUX. Compt. rend., 152, 1860-2.—The adherence of Pb arsenate is generally great on glass and on vegetable epidermis. The age of the suspension does not appear to have any effect on its adherence to glass and only affects slightly the adherence to the vine. When 10-20 days old, the suspension has about <sup>2</sup>/<sub>4</sub> the adhesive power of that prepared 5 days before use.

M. X. Sullivan.

Report on Insecticides. C. C. McDonnell. U. S. Dept. Agr., Bur. Chem., Bull. 137, 36-40.—A report of cooperative work on methods for detg. total and water sol. As and total Pb in Pb arsenate and total As<sub>2</sub>O<sub>2</sub> and As<sub>2</sub>O<sub>3</sub> in London purple.

C. C. McDonnell.

German Potash Industry (Krische). 18. Superphosphate in France (G. A. Br.). 18.

HART, E. B. AND TOTTINGHAM, W E: General Agricultural Chemistry. Madison, Wis.: 334 pp., \$1.50.

Heim, G.: Der deutsche Kalibergbau und die Kaligesetzgebung. Regensburg: G. J. Manz. 43 pp., 50 M.

HÖPPNER, G.: Die Kunstdüngermittel und ihre Anwendung in der modernen Landwirtsdhaft. Leipzig: Hans Wehner. 8°, 255 pp., 2.50 M.

INGLE, H.: Manual of Agricultural Chemistry. Scott, Greenwood & Son. 450 pp., 2.25.

Brit., 18,523, Aug. 5, 1910. G. C. TARANTO, Italy. Manuf. of dry non-acidulated superphosphates, the sol. of the phosphoric acid of which in H<sub>2</sub>O is increased by opening up mineral phosphate by a mixt. of H<sub>2</sub>SO<sub>4</sub> and HNO<sub>3</sub>, by mixing the phosphate with about 99% of the quantity of H<sub>2</sub>SO<sub>4</sub> generally used, to which has been added 1-5% of conc. HNO<sub>3</sub>.

## 16. FERMENTED AND DISTILLED LIQUORS.

#### ROBERT WAHL.

Characterization of Rose Colored Wines and White Wines from Red Grapes. J. LABORDE. Ann. jals., 4, 389-91.—Polemical. The differentiation is based on a study of the tannins present in the wines.

C. P. WILSON.

Interpretation of Analytical Results and the Valuation of Wine. Anon. Ann. Jals., 4, 356-61.—Where the source of a wine is indicated it is easy to judge the wine by comparing it with other wines known to be produced in the same region. When this information is not available a wine is to be judged by: (A) the actual amt. of the constituents present; (B) the ratio alc.: ext. which is from 2.5 to 4.5 in red and from 3.5 to 6.5 in white wines; (C) sum of alc. plus total acid which should be from 13 to 17%, falling in exceptional cases to 11.5%; (D) Halphen's ratio, R = fixed acid plus 0.70 ÷ % alc. This ratio for wines of 6 regions is plotted as curves; (E) Roos ratio, E = G/D, C and D being the values obtained for (C) and (D) above. For red wines E = 3 to 3.2 and for white wines should be not less than 2.4. Spoiled wines are easily known by the presence of the microörganisms which cause the spoilage.

C. P. WILSON.

The New Official Bavarian Saccharimeter. C. Wirth. Z. ges. Braww., 34, 200-3.

—Tests made have shown that the officially graduated thermo-saccharimeter at 17.5° indicates 0.1-0.2% more ext. in a wort than the corresponding pycnometer detn. Solns. of NaCl and of pure sugar do not display this discrepancy. From these facts the author concludes that the above variation is due to the different physical behavior, especially surface tension, of the sugar solns. and of the wort.

C. A. Nowak.

Spanish Wines Imported into France. FILANDEAU. Ann. jals., 4, 362-80.—
The wine districts of Spain and analyses of wines from 7 regions are discussed. Spanish wines are richer than French wines and cannot be judged by the same standards. The ratio of alc. to ext. is noticeably high.

C. P. WILSON.

The Determination of the Volatil Acids in Wine. A. Verda. Schweiz. Wochschr., 49, 340-1.—The volatil acids were detd. by passing steam thru 50 cc. wine until 200 cc. of distillate were obtained and then titrating. An Italian wine was found to yield acid up to 400 cc. of distillate; it contained lactic acid, but whether this was the cause of the abnormality was undetd.

H. C. Fuller.

The Problem of the Estimation of the Acids in Wine. A. QUARTAROLI. Viterbo. Chem. Lab. of the Tech. Inst. Staz. sper. agrar. ital., 44, 138-40; thru Chem. Zentr., 1911, II, 54.—C. von der Heide and W. J. Baragiola (C. A., 5, 2691) have quite misunderstood the author's method (C. A., 5, 2297).

J. A. LE CLERC.

Possible Forms of Phosphorus in Grapes and in Wine. J. VENTRE. Annal. ecol. nat. agr. Montpellier, 10, 1-103; thru Zentr. Biochem. Biophys., 11, 341.—In grapes and in wine P exists as inorg. P and in org. combinations sol. in Et<sub>2</sub>O and absolute EtOH. In the stem the P is found joined to carbohydrates or as acids of P. The same forms are found in the skin. Lecithin, which is present in the skin, gives palmitic and stearic acids on decomp. H<sub>2</sub>PO<sub>4</sub> and choline are found in the must. The lees contain lecithin. Glycerophosphoric acid and diethyl phosphoric acid and possibly other P comps. occur in the wine also.

I. K. Phelps.

Enological Studies. WILLIAM B. ALWOOD. U. S. Dept. Agri., Bur. Chem., Bull. 140.—The results are given of the exam. of a number of samples of one seedling and of the Pocklington and Worden varieties, showing that sucrose is a constituent of those varieties in an amt. varying from 2 to over 10%. The sugar and acid content of a no.

of varieties of American grapes was detd. at various intervals during ripening and at full maturity.

W. D. Bigelow.

The Bactericidal Action of Wines and Alcoholic Drinks. A. T. Gaillard. Lausanne. Mitt. Lebens. Hyg., 2, 40-64, 124-60; thru Chem. Zentr., 1911, I, 1871-2.— The work of Munier, Seiler, and Roux (cf. C. A., 4, 1080) was continued, using most of the common alc. drinks. In general, it was found that (1) the number of bacteria in water decreased about 95% upon the addition of alc. liquids in amts. depending on the % of alc.; (2) about 30 species of bacteria, chiefly non-pathogenic, survived the expts.; (3) the bactericidal action is greatly increased by the presence of fruit acids, the max. action occurring when alc. and acid are present in amts. agreeing with the alcohol-acid rule of Gautier or Masure; (4) in drinks containing less than 15% alc., the action of the acids is at least as strong as that of the alc.; and (5) the presence of essences did not appear to increase the bactericidal action. M. Heidelberger.

The Juice of the Beer Yeast. E. KAYSER. Compt. rend., 152, 1279-80; cf. C. A., 5, 2144.—The most active zymase preps. were obtained by allowing beer yeast to remain for 35-40 hrs. at 25° before treating according to Lebedev's process. The action of the ferment thus obtained is increased by salts of Mn as is yeast itself.

H. A. SPOEHR.

The Influence of Humates on Microbrganisms. E. Kayser. Compt. rend., 152, 1871-3.—Humates of NH, added to peptone sugar bouillon increased the formation of organic acids from the various sugars. Added to sugared wort it had no effect on yeast from cider while it increased the activity of wine yeast. Its action seems to be due to the nutrient it adds.

M. X. Sullivan.

Thujone in Absinthe. H. Enz. Schweiz. Wochschr., 49, 337-40.—The red coloration which thujone gives with Na nitroprusside and AcOH is also obtained with aldehydes, but the influence of the latter may be avoided by first treating the distillate with 10% ZnSO4. It has been recommended to fix the aldehydes with aniline phosphate, but there is always a small amt. which remains. This may be avoided by carrying out a fractional distillation, EtCHO coming over in the first two, thujone in the 6th and 7th, and the high boiling ethereal oils, anise and fennel in the last. The red reaction is given by the oils of hyssop, calamus, verbena, savin and also carvone and citral.

H. C. FULLER.

The Relation between the Actual and the Calculated Beer-wort Extract. Schonfeld. Z. angew. Chem., 23, 984.—In the literature, the values for the extractive content of the beer wort as calc. by Balling's formula, are lower (about 5%) than the actual. This deviation has been connected with alc. evapn. during brewing. The author has performed expts. which show that in the cases of bottom fermentation and cold top fermentation the alc. evapn. is not a factor (calc. values higher than actual). In other cases where higher temps, are required the evapn. should be considered. Some calculated results deviate from the actual due to too high a yeast factor in the formula.

Geo. W. Stratton.

Cooperation in Experiments on Barley, Color Malt and Caramel Malt. Wolfs. Nürenberg. Z. angew. Chem., 23, 983.—The author offers suggestions for uniform methods of analyses.

G. W. STRATTON.

Experiments Concerning Several Mechanical and Thermal Effects on the Water and Extract Value of Malt. A. WLOKKA. Z. angew. Chem., 24, 1405-10.—The varying methods and results of malt analysis obtained by diff. Scientific Stations are the cause of much unjust annoyance to the maltster. The author emphasizes the necessity of reducing the limits of error in malt analysis and, based upon his work, makes the following suggestions to be considered in the contemplated revision of

standards: 1. Only 1 uniform method of detg. moisture should be used in all detns.

2. Time of drying should be fixed at 3 hrs. from the time the sample is introduced into the oven previously heated to  $104-5^{\circ}$  C. During drying the temp. should remain constant at this point for at least  $2^{1}/_{2}$  hrs. 3. Adoption of a uniform app. for drying as regards size, draught, etc. 4. Fixing of standards for dimensions of drying glasses for use in the Scholvien app. 5. Provided the above are carried out the limits of error in detg. moisture can be reduced to 0.15-0.20%. 6. For mashing app. with propeller stirrers the no. of revolutions should be fixed, preferably, at a minimum of 90 per min.

C. A. Nowak.

The Bitter Acid Content of Bavarian Hops of the Season 1910. F. WAGNER. Z. ges. Brauw., 34, 161-3, 173-7.—The author reports a number of analytical detns. of the bitter substances in hops. He points out that in general the results agree with those obtained by the customary mechanical exam. Detn. of the bitter substances alone should never be considered as sufficient for the valuation of a sample. Age, method of drying, preserving, and storing, must be given equal attention.

C. A. NOWAK.

The Manufacture of Alcohol from Sugar Beets. K. Antal. Z. Spiritusind., 34, 239-40, 252-3.—Owing to the falling off of potato crops, sugar beets have in recent years been employed in Austria-Hungary as a substitute in the production of alc. It is now possible to use the same plants that were originally built for potatoes. The author gives the working details of these plants in which the beets are extracted with steam under pressure in a Henze converter. When potatoes and beets are worked together, potato, lactic acid, or sulfuric acid yeast is employed in fermentation. The results so obtained are, however, not as satisfactory as if the potatoes and beets are steamed separately. While in fermenting beet mashes alone pressed beer yeast is sometimes employed (15-20 l. to 1000 1-mash) pure culture wine yeast capable of fermenting sucrose and raffinose is preferable. Using wine yeast, 60.4 l. of alc. are produced per 100 k. of sugar while beer yeast yields only 54-55 l. Inasmuch as the distillation residue is used as fodder, the use of beets compares favorably with that of potatoes from an economical standpoint.

C. A. Nowak.

Alcoholic Fermentation (LEBEDEV). 11.

Analysis of Soft Drinks (Fuller). 12.

Ethereal Oils (SEMMLER, MAYER). 10.

Hop Blossom Oil (BECKMANN, DEUSSEN). 10.

BARAGIOLA, W. J.: Weinfälschung. Verlag der Schweizer Wein-Zeitung. 24 pp. NORTON, C.: Modern Yeasting and Distillation. Chicago: Mitchell, Larimer & Titus. 8°, \$12.00.

Brit., 9,777, Apr. 21, 1910. E. W. Kuhn, 133 Tooley St., London Co. Etherification and clarification of fermented beverages by preparing wort having a predetd. % of glucose, then fermenting it with yeast in closed vats at atm. pressure, then sterilizing the fermented liquor under high pressure, and etherifying it in vats out of contact with the air by means of a bacterial culture, then transferring it into a jacketed cooling tank under a counter pressure of filtered sterilized air, hermetically sealing the tank and introducing a further quantity of glucose and yeast for the production of CO<sub>2</sub> by fermentation of the glucose, the liquor being then cooled.

Brit., 16,707, July 13, 1910. C. R. WAGNER, 14 Gartenstrasse, Sonnenberg bei Wiesbaden, Ger. Mfg. alcohol-free drinks from wine, beer, wine made from fruit

and the like, containing the aromatic substances of the initial liquids, by fractional distillation of these liquids in vacuum vessels and sepg. out the alc. carried over by fractional cooling as well as by absorption of the various fractions of the aromatic substances by suitable mixts. of liquids and combining the latter with the residue of distillation, the various fractions obtained by corresponding detn. of the temps. and vacuum, being removed directly after the production from the action of the vacuum in the app., the fractional distillation being itself carried out so that in the first stage only the most volatil aromatic substances are evapd. and absorbed in the first absorption vessel, whereas in the second stage, from the mixt. of the less volatil aromatic substances and of the alc. vapor thus obtained, the latter is sepd. out by fractional cooling and the aromatic substances are absorbed separately in the second absorption vessel, and finally in the third stage the alc. still present is distilled over.

Brit., 19,024, Aug. 12, 1910. A. GARRAUD, 10 rue de Metz, Cognac, France. Improving or artificially aging wines, spirits and liquors, by producing a mol. agitation within the liquid to be treated by means of Hertzian waves generated by wave producing app. preferably immersed in the liquid.

Brit., 20,063, Aug. 29, 1910. ALFRED LAMBERT, 12 St. James Square, London, S. W. Drying and coloring malt by feeding the malt thru zones of progressively increasing temp. while causing fresh surfaces of the malt to be exposed to the air, and regulating the temp. in the various zones, so as first to dry and finally to color the malt.

Brit., 5,346, Mar. 3, 1911. MUHLENBAUANSTALT, Dresden A 7, Ger. In the separation of malt into meal, semolina, and husks, the pure husk particles are removed by the action of a continuously moving air current, from the husks obtained by sifting, and if desired also from the semolina, and then again added to the other pure husks only obtained after another grinding and sifting, during or after the sifting.

### 17. PHARMACEUTICAL CHEMISTRY.

### V. COBLENTZ.

The Advancement in the Pharmaceutical-chemical Industries in 1910. RICHARD LUDERS. Chem. Ind., 34, 148-56, 182-8, 212-8, 243-8. E. J. CRANE.

Hydrolysis of "Iodeigon-sodium." ADOLF OSWALD. Agr. Chem. Lab., Polytechnikum, Zurich. Z. physiol. Chem., 72, 374-9.—"Iodeigon-sodium," is an iodoprotein prep. put up by the Chem. Fabrik Helfenberg containing 15% I. This prep. differs from other iodoproteins in that it does not yield diiodotyrosine on analysis. All but about 3% of its I is obtained as HI.

G. M. MEYER.

The Estimation of Minute Quantities of Nitroglycerin. W. L. Scoville. Am. J. Pharm., 83, 359-64.—An alc. soln. of nitroglycerin, containing approx. 0.00065 g. is evapd. spontaneously, treated with 2 cc. phenoldisulfonic acid soln., rendered slightly alkaline with KOH, dild. to 100 cc. and compared in a colorimeter with 1.2 cc. of a standard soln. of KNO<sub>2</sub> equiv. to 0.00065 g. nitroglycerin treated in the same way. In the case of  $\frac{1}{100}$  grain tablets, 5 are powdered, shaken 2 hrs. with 10 cc. EtOH, filtered, and 2 cc. used for the test. Tablets of other strengths are treated similarly, the equiv. of  $\frac{1}{100}$  grain being taken for the test.

Variations in the Form of Digitalis Hairs. H. KRAEMER. Am. J. Pharm., 83, 365-70.—Digitalis contains 2 types of hairs, glandular and non-glandular, and in different specimens one or the other will often predominate, the glandular usually occurring in greatest number in the cultivated leaf. It would be of interest to det.

whether there is any parallel between the physiological activity and the type of hair.

H. C. FULLER.

The Preparation of Neutral Suspensions of Salvarsan. G. M. Beringer, Jr. Am. J. Pharm., 83, 371-3.—The ampule is opened and the salvarsan emptied into a mortar, 15 drops of 15% NaOH added and H<sub>2</sub>O drop by drop to make a thin paste; if not faintly alkaline NaOH is added and the whole thoroly triturated; dil. HCl is then added until the mixt. just ceases to react, H<sub>2</sub>O added to 8 cc., poured back into the ampule, the mortar washed with 2 cc. H<sub>2</sub>O, and the ampule sealed.

H. C. FULLER.

A New Vegetable Adulterant. H. Kraemer. Am. J. Pharm., 83, 377-81.— A macroscopic and microscopic description of the outer layers of the pericarp of the fruit of Juglans regia, English walnut, the powder of which is being imported probably to replace ground olive pits as an adulterant. Its stone cells resemble those of olive pits; wi h H<sub>2</sub>O and alkali a dark reddish brown color is obtained similar to that given with black pepper hulls and black walnut and pecan shells.

H. C. Fuller.

A Reaction for Caulophyllin. J. F. H. GILBARD. Analyst, 36, 270–1.—5 cc. of an alc. ext. obtained by digesting 0.1 g. caulophyllin in 25 cc. 80% EtOH is evapd. to dryness in a porcelain dish, the residue is taken up with water, broken up with a flatheaded rod, 2 cc.  $H_2SO_4$  are added and well stirred when an intense purple or reddish purple color is produced in 5 min. In examining a pill 0.1 g. is repeatedly extd. with 80% EtOH, evapd. and the residue treated with  $H_2SO_4$ ; if caulophyllin is present a distinct blue color will be shown at the edges after a few mins.

H. C. FULLER.

Some Supplementary Facts Regarding the Physiological Testing of Digitalis Leaves. Focke. Arch. Pharm., 249, 323-8.—In the prep. of a digitalis infusion it is immaterial whether a cotton or linen filter is used; the strength is the same. 10% infusions in EtOH or H<sub>2</sub>O hold their strength as well as one of lesser %. H<sub>2</sub>O extracts from the leaves about 85% of the active principles.

H. C. Fuller.

A Short History of Bromine, the Bromides and other Bromine Compounds Employed in Medicin. G. Sharp. Pharm. J., 87, 128-30. H. C. Fuller.

Official Ointments, with Special Reference to the Substances Used as Bases. R. B. WILD. Pharm. J., 87, 131-3.—Soft paraffin and paraffin ointment appear to be hardly absorbed at all, lard and olive oil are absorbed to a considerable extent (15% in 2 min.), and hydrous wool fat 20% provided the proper amt. of H<sub>2</sub>O is present. Classified according to base the official ointments should be divizible into (a) protective, when the effect is desired to be on the epidermal surface only; (b) emollient, when the deeper parts of the skin are to be reached; (c) absorbent, when it is desired to secure a passage thru the skin into the body. For (a) soft paraffin is a good base tho it runs easily in warm weather and is without sufficient body for certain purposes unless 1 pt. each of starch and ZnO is added to 2 pts. soft paraffin. The official rosewater ointment makes a good emollient base but is difficult to make and does not keep well owing to sepn. of H<sub>2</sub>O. Equal parts of hydrous wool fat and "glycerin of starch" form a useful base. For absorbent ointments lard and hydrous wool fat fulfil all purposes. Ammoniated Hg ointment should be reduced to 5% and the base should be lard or simple ointment; red HgO ointment should be made up with the same base. The present pptd. Hg oleate is considerably less active and penetrating than that made by combining HgO and oleic acid. Pb(OAc), should be combined with wool fat and H. C. FULLER. soft paraffin instead of glycerol.

Further Note on Podophyllum emodi. J. C. UMNEY. Pharm. J., 87, 156-7.— A supply of the rhizome collected after fruiting contained about twice as much podophyllotoxin as P. pelialium.

H. C. Fuller.

The Supposed Loss of Morphine in the Preparation of Tincture of Opium. E. H. FARR AND R. WRIGHT. Pharm. J., 87, 158-60.—When the official methods are followed there is always a loss of 0.8-9.0% of morphine varying owing to incomplete extraction.

H. C. Fuller.

Extract of Indian Hemp. H. Deane. Pharm. J., 87, 160-1.—Exts. obtained from Madagascar cannabis contain a larger amt. of resin. The ext. obtained by the Brit. Pharm. process is unsatisfactory; it contains a brown extractive which tends to cause sepn. and, being somewhat insol. in EtOH, gives a poor tincture. A better extract results if the brown extractive is washed away with H<sub>2</sub>O, and it would be well if this change could be made in the official extract as both products are at present sold on the market.

H. C. Fuller.

Note on Spirit of Sal Volatii. E. W. Pollard. Pharm. J., 87, 162-3.—The official process is not practicable for the av. pharmacist and a spirit equal to it in all respects may be obtained by dissolving 4 oz. (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> in 8 oz. NH<sub>4</sub>OH 28% and 9 oz. H<sub>2</sub>O and then adding 7 pints of a mixt. prepared by distilling 4.5 drams oil nutmeg, 6.5 drams oil lemon and 2 pints H<sub>2</sub>O to 1 pint to which is added 6 pints EtOH.

H. C. FULLER.

A Suggested Standard for Thyroidium Siccum. R. R. BENNETT. Pharm. J., 87, 163-4.—The av. amt. of I was found to be 0.158% and as this figure is the same as found by other workers it is suggested that an I stand. of 0.15% be adopted.

H. C. FULLER.

Secret and Proprietary Medicins. E. P. HARRISON. *Pharm. J.*, 87, 165-8.—
A discussion of the extent of the sale of patent and proprietary medicins in England and a resolution passed at the Brit. Pharm. Conference.

H. C. FULLER.

Linimentum Ammoniae. F. H. ALCOCK. Pharm. J., 87, 171-2.—Instances of solidification of the Brit. Pharm. liniment are noted and it is suggested to modify the formula, using 2 oz. olive oil and 1 oz. stronger NH<sub>4</sub>OH.

H. C. FULLER.

Note on Spirit of Nitrous Ether. D. B. DOTT. Pharm. J., 87, 173.—It is proposed to have 2 solns., one of NaNO<sub>3</sub> and the other of EtOH containing lactic acid, which can be mixed as required, thereby avoiding the loss which takes place in ordinary spirits.

H. C. FULLER.

Note on Strychnine Hypophosphite. D. B. Dorr. Pharm. J., 87, 173.—BH<sub>2</sub>PO<sub>2.3</sub>H<sub>2</sub>O is sol. in H<sub>2</sub>O (1-3.3) and becomes anhydrous at 100°. H. C. F.

Note on Arsenates of Strychnine. D. B. Dott. Pharm. J., 87, 173.—B<sub>2</sub>H<sub>2</sub>AsO<sub>4</sub> is sparingly sol. and partly decomposed in H<sub>2</sub>O, BH<sub>2</sub>AsO<sub>4</sub>.2H<sub>2</sub>O is well recognized, sol. in H<sub>2</sub>O (1-34) and rendered anhydrous at 120°. B(H<sub>2</sub>AsO<sub>4</sub>)<sub>2</sub>.H<sub>2</sub>O is sol. (1-16.75) but is little known and the strychnine content is small in comparison. H. C. F.

The Moisture Content of Medicinal Extracts. K. C. ALLEN AND T. BREWIS. Pharm. J., 87, 172-3.—The amt. of moisture in the solid ext. of a certain drug is not a constant quantity; in fact the directions for the prep. of an extract admit of wide interpretation and lead to uncertainties with regard to the physical condition. Extracts standardized on the basis of the crude drug taken where there is no potency based on chemical or physiological methods, should have a standard based on the really dry extractive actually obtained. Soft extracts, gentian, taraxacum, etc., and dry extracts, aloes, cascara, etc., should have definit limits of moisture prescribed. Tables give the moisture and ash contents of a number of extracts.

Note on Solution of Sodium Ethylate. H. FINNEMORE. Pharm. J., 87, 174-5.

—Sodium ethylate darkens and while this does not effect its value as a therapeutic agent, it is unsightly. Na methylate has the same valuable properties, it never becomes dark and may be substituted.

H. C. FULLER.

An Experiment in Peppermint Culture. H. J. HENDERSON. Pharm. J., 87, 175-6.—Plants grown in the shade contained much less oil than those exposed to the sunlight.

H. C. FULLER.

The Constitution of Commercial Bismuth Subchloride. J. B. P. HARRISON. *Pharm. J.*, 87, 179.—When prepared according to the methods of the Brit. Pharm. codex this salt has the comp. BiOCl, but when made by the subnitrate with a soln. of NaCl its constitution in general will be  $Bi_{13}O_{13}Cl_{10}$ .

H. C. FULLER.

Note on Bartsia odontites. H. Finnemore and G. E. Town. *Pharm. J.*, 87, 180.—An extract was freshly made, tested physiologically and found to be inert.

H. C. FULLER.

White Precipitate and the Analysis of White Precipitate Ointment. G. D. ELSDON. Pharm. J., 87, 180-1.—Hg in the salt was detd. as HgS, weighing in a Gooch crucible. The com. article contains about 77-78% Hg. The amt. of salt in the ointment was detd. by treating the sample in a separator with petroleum and HCl and pptg. the acid soln. with H<sub>2</sub>S. The character of the base may be detd. by treating the ointment with H<sub>2</sub>SO<sub>4</sub> and warming, when the presence of other bases than paraffin will be noted by charring. If another base is suspected an I no. is detd. H. C. FULLER.

Report on Medicinal Plants and Drugs. G. W. HOOVER. U. S. Dept. Agri., Bur. Chem., Bull. 137, 181-3.—The results obtained by the present pharmacopeial methods for detg. the various alkaloids in potent drugs are fairly satisfactory. All known methods which have been employed for the detn. of morphine have been tried and a shake-out method with CHCl<sub>2</sub>-EtOH has been modified so that it gives satisfactory results. The work on crude drugs shows that the language employed in describing the physical standards should be more specific.

H. C. FULLER.

Report on Headache Mixtures. W. O. EMERY. U. S. Dept. Agri., Bur. Chem., Bull. 137, 183-6.—The results of cooperative work on the analysis of mixtures containing caffeine, acetaniline, NaHCO<sub>3</sub>, sugar, etc., were very satisfactory, but those obtained with the acetophenetidine mixture were less uniform. In the latter case the difficulty is due to the treatment with  $H_2SO_4$ ; 10 cc. should be used and the heating on the bath should cease when  $\frac{1}{2}-\frac{1}{2}$  has evapd. Salicylates often occur in these mixtures simultaneously with boracic, benzoic or cinnamic acids, and may be estd. by a modification of Bougault's method; the sample containing approx. 0.1-0.2 g. salicylic acid is dissolved in 100 cc.  $H_2O_1$  1-2 g. NaHCO<sub>2</sub> added, heated nearly to boiling, 35-70 cc. 0.2 N I in KI added, heated on the steam bath 1 hr., the pptd. tetra-iododiphenylenequinone filtered onto a tared gooch, washed with  $H_2O_1$  dried and weighed (see C. A., 3, 2344; 4, 2351).

A Simplified Extraction Method for the Determination of Morphine in Opium and Opium Preparations. E. O. EATON. U. S. Dept. Agri., Bur. Chem., Bull. 137, 188-9.

—I g. of the dried and powdered sample is shaken with 100 cc. lime water, 50 cc. filtered into a separator and shaken with CHCl<sub>2</sub> and Et<sub>2</sub>O, the solvents then being washed with lime water which is added to the original 50 cc.; 20 cc. EtOH are then added, 1% NH<sub>4</sub>Cl until NH<sub>3</sub> is freely given off, followed by 30 cc. CHCl<sub>2</sub>. The mixt. is shaken, the CHCl<sub>3</sub> drawn off and the shaking repeated with 4 successive portions of 30 cc. each CHCl<sub>3</sub>-EtOH 4-1, the combined solvents are washed, filtered, evapd. in a tared dish and the residue titrated as morphine. For paregoric 100 cc. are evapd. to 15 cc., transferred to a separator and treated as above. For soothing syrups 100 cc. are acidulated, NH<sub>4</sub>OH added in excess, extd. with CHCl<sub>3</sub>-EtOH mixt. to total exhaustion, the solvents evapd., dissolved in lime water and treated as above.

H. C. F.

Progress in Microchemical Tests for Alkaloids. B. J. HOWARD AND C. H. STE-PHENSON. U.S. Dept. Agri., Bur. Chem., Bull. 137, 189-90.—The crystallin ppts. of

67 alkaloids with 40 different reagents have been studied microchemically, photographs of over 200 compds. have been prepared, and a study is being made of the effect of one alkaloid on the cryst. form of the ppt. of another. It is of interest to note that the glass rod used in starting cryst. has considerable influence on the test, a new rod often producing no result while one previously used with the same reagent and alkaloid causing immediate ppt.

H. C. FULLER.

Analysis of Beef, Iron and Wine. E. A. RUDDIMAN AND L. F. KEBLER. U. S. Dept. Agri., Bur. Chem., Bull. 137, 194-7.—Beef, iron and wine made according to the N. F. should possess the following characteristics, 50 cc. when assayed by the Gunning method for total N should yield not less than 0.10 g. N coming from the beef ext., contain not less than 0.07 g. nor more than 0.08 g. Fe, and the alc. should not be under 14 nor over 20%. If the amt. of N obtained by the BaCO<sub>3</sub> method exceeds 0.006 g. the excess is to be considered as coming from an NH<sub>4</sub> compd. and is to be deducted from the total N, the remainder representing the N coming from the beef ext. The difficulty of estg. N of NH<sub>4</sub> compds. in the presence of ext. of beef is due to the fact that the latter contains bodies that are easily decomposed forming substances that act like NH<sub>3</sub>, hence any method which would liberate all the NH<sub>3</sub> in a compd. like Fe and NH<sub>4</sub> citrate would also decompose some of the N compds. in the beef. To det. the N in the NH<sub>3</sub> liberated by BaCO<sub>3</sub> 50 cc. of the sample are introduced into a Kjeldahl flask, 200 cc. H<sub>2</sub>O added, 3 g. BaCO<sub>3</sub>, and granulated Zn or pumice; connection is made with a condenser and the distillate collected in 0.1 N H<sub>2</sub>SO<sub>4</sub>.

H. C. FULLER.

A Mandragora Root. C. HARTWICH. Schweiz. Wochschr., 49, 269-73.—An undetd. root, obtained from Smyrna and resembling ginseng in form was sold as mandragora. It contained considerable starch similar to that found in belladona.

H. C. FULLER.

Preparation of Potions of Menthol. A. Andemard. Schweiz. Wochschr., 49, 341-2.—When menthol cannot be administered in the form of an alc. soln. it may be dispensed in aq. soln. in the form of an emulsion using mucilage of acacia or Irish moss.

H. C. Fuller.

Concerning Newer Remedies and their Meaning for Modern Therapy. LIPOWSKI. Bromberg. Münch. med. Wochschr., 58, 358-60.—Chemical classification and discussion of efficiency.

G. R. HENRY.

Certain Physical and Physiological Properties of Stovaine and its Homologs. V. H. Veley and W. L. Symes. Physiol. Lab., Univ. London. Proc. Roy. Soc. London, (B) 83, 413-20.—Me, Et, amyl, phenyl, and benzyl stovaines had the following: d<sub>16</sub> respectively 1.213, 1.207, 1.106, 1.191, and 1.178. The affinity value of Fourneau's salt for cocaine, stovaine, and Me stovaine were detd. as well as some physiological effects.

I. K. Phelps.

The Constituents of Fagara zanthoxyloides Lam. Hans Priess. Ber. pharm. Ges., 21, 227-67.—The material was obtained from German Togo, E. Africa. I. The fruit consisted of the peel which surrounded the black glistening seed. These latter contained 31.9% of fatty oil. An ethereal oil amounting to 2.4% was obtained from the fruit by steam distillation and from this on cooling to  $-5^{\circ}$ , 3-4% of a cryst. N-free substance sepd., m. 144°, C = 66.55%, H = 3.53%. The original clear oil soon darkened. Its constants are:  $d_{15} = 0.9229$ ,  $[\alpha]_{15}^{16} = -1.20^{\circ}$ , acid no. 2.19, ester no. 58.51, sapon. no. 60.70. Analysis gave C = 82.1%, H = 10.5%, O = 7.4%. Among the compounds obtained by various reactions from this oil were methyl-n-nonyl ketone and its semicarbazone, caprinic acid and its amide, m. 98°, AcOH acid, dipentene, linaloöl, and an unidentified sesquiter pene. The residue of fruit peels remaining after

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removal of the ethereal oil was extracted with hot alc. and a N-free cryst. lactone called zanthotoxin,  $C_{12}H_aO_4$ , m.  $144-5^\circ$  obtained. Excess of alkali gives a salt which in neutral or alkaline soln. gradually decomposes into free alkali and the original lactone. It is an active fish poison. The bark of the roots of the plant by  $C_aH_a$  extraction gave 1% of jagarol,  $C_{20}H_{12}O_a$ , cryst. and m.  $127-8^\circ$ . II. A pharmacognostic description of the plant, also a table of varieties of Fagara and Zanthoxylum which have so far been studied.

A. Seidelle.

"Busch Salt." W. Lenz. Ber. pharm. Ges., 21, 270-2; cf. C. A., 5, 765.—A new sample of this material contained SiO<sub>2</sub> 0.25%, Al<sub>2</sub>O<sub>8</sub> + Fe<sub>2</sub>O<sub>3</sub> 0.23%, K<sub>2</sub>SO<sub>4</sub> 18.48%, KCl 77.77%, H<sub>2</sub>O 2.29%, insoluble 0.35%.

A. SEIDELL.

Medicinal and Useful Plants of Brazil. TH. PECKOLT. Ber. pharm. Ges., 21, 273-9; cf. C. A., 5, 1494.—The Combretaceae are described.

A. SEIDELL.

The Identification of Creosote and Guaiacol Carbonates. A. FERNAU. Z. Oesterv. Apoth. Ver., 49, 165.—The sample is saponified in an open dish on the steam bath with alc. KOH, the soln. acidified, shaken after addition of NaCl and the vol. of the liberated creosote or guaiacol measured.

A. SEIDELL.

Plant Lactones as Fish Poisons. HANS PRIESS. Ber. pharm. Ges., 21, 267-70.

—The characteristics of these poisons are described.

A. S.

Action of Combination of Drugs (BURGI). 11.

Analysis of Soft Drinks (FULLER). 12.

An Animal Alkaloid (AVERKIEV). 11.

Determining Ether Extract (LEACH, HILTNER). 7.

Ethereal Oils (SEMMLER, MAYER). 10.

Hop Blossom Oil (BECKMANN, DEUSSEN). 10.

Oil from St. Geran (STRIYOV). 22.

Properties of Polyporaceae (FORD, SHERRICK). 11.

Purity of Foods and Drugs (TANKARD). 12.

Quinine and Euquinine (ASTRUC, COURTIN). 10.

Soaps from Different Glycerides (HAMILTON). 27.

Terpenes and Ethereal Oils (WALLACH). 10.

Thujone in Absinthe. 16.

Use of Antimony Compounds (ROWNTREE, ABEL). 11.

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Brit., 5,931, Mar. 9, 1910. Wm. H. PERKIN, The University, Manchester. Mfg. compounds of the type of  $\alpha,\beta,\beta$ -trimethyl- $\beta$ -hydroxypropionic ester, by subjecting a mixt. of a ketone and an  $\alpha$ -halogen fatty ester to the action of Zn and then of  $H_2O$ .

Brit., 15,187, June 25, 1910. Mrs. Susan Twigg, Horn's Hotel, Farnworth, near Widnes. A remedy for the treatment of skin diseases, consisting of hog's lard 1 lb., S 1 lb., camphor 4 oz.

Brit., 15,649, June 29, 1910. BAD. ANILIN- & SODA-FABR., Ludwigshafen a/R, Ger. Mfg. condensation products of the anthracene series by reacting on a halogenated or nitrated dianthraquinonyl compd., with a salt or an ester of an o-aminoarylcarboxylic acid of the benzene series. Coloring matters of the anthracene series contg. 1 or more than 1 acridone ring are obtained by treating with a condensing agent the condensation product above mentioned.

Brit., 17,356, July 21, 1910. FARBEN. VORM. F. BAYER & Co., Berlin, Ger. Production of pharmaceutical compounds by treating oxymercuric carboxylic acid, anhydrides, or their derivs., with NH<sub>2</sub> or primary, secondary, or tertiary amines of the fatty series and secondary amines such as piperidine, and either with amino acids or their salts, or with such nitrogenous compds. as, while being neutral to litmus have at the same time both basic and acid character or their salts, or with imino compds. contg. groups of an acid character, or by treating the double compds. obtainable from the oxymercuric compds. and NH<sub>2</sub> or amines with the said nitrogenous compd. Cf. C. A., 5, 2905, 2906 and 2907.

Brit., 22,097, Sept. 23, 1910. Idem. Producing sulfaminobenzoylaminonaphthols or sulfaminobenzoylaminonaphtholsulfonic acids or derivs. of both these compds. substituted in the benzoyl group, by treating nitrobenzoylaminonaphthols or nitrobenzoylaminonaphtholsulfonic acids or derivs. of both these compds. substituted in the benzoyl group, with neutral or acid salts of sulfurous acid.

Brit., 24,690, Oct. 24, 1910. FARBWERER VORM. M. L. & B., Höchst a/M, Ger. In the process of isolating alkali salts of indoxyl or its homologs or derivs. substituted in the benzene nucleus described in 22,288, of 1910, operating with melts of bodies which contain a carboxylic group attached directly to the benzene nucleus and yield indoxyl or indoxyl carboxylic acid or homologs or derivs. thereof substituted in the benzene nucleus when fused with alkali.

Brit., 25,873, Nov. 7, 1910. H. S. Wellcome, Snow Hill Bldgs., London. Mfg. a therapeutic compound by the action of KSCN on diaminoacetone dihydrochloride, the product being a basic substance, 2-thiol-4-(or 5)-aminomethylglyoxaline. Details are specified. The reaction proceeds in the sense of the following equation:

$$\begin{array}{cccc} CH_1 \cdot NH_2 \cdot HCI & C \cdot NH \\ CO & + KSCN = C \cdot N = C \cdot SH \\ CH_2 \cdot NH_3 \cdot HCI & CH_3 \cdot NH_3 \cdot CI \end{array}$$

Brit., 28,378, Dec. 6, 1910. BLIEBERGER, WEBER & Co., 278 W. 71st St., N. Y. Mfg. menthylated methylic ester of salicylic acid or the menthylated wintergreen oil. See C. A., 5, 972.

Brit., 28,538, Dec. 8, 1910. H. S. Wellcome, Snow Hill Bldgs., London. Mfg. a physiologically active base according to the following scheme:

Brit., 29,165, Dec. 15, 1910. O. L. A. Dubosc, 68 rue Caumartine, Paris. Manuf. of formates by the action of moist CO on a methylamine or pyridine, in the form of vapor, in the presence of an inert mass, impregnated or not with a catalytic agent such as Cu, Ti, V, Ni, Fe, Pt, or an oxide or a salt thereof, the reaction being effected at atm. pressure and at a temp. between 90 and 165°.

Brit., 29,907, Dec. 23, 1910. J. A. von Wulfing, 231 Friedrichstrasse, Berlin. A therapeutic preparation is obtained by combining with cane sugar 5 mols. HCHO for each mol. of cane sugar, and with monoses such as glucose, of 5 mols. HCHO for each 2 mols. of monose.

Brit., 2,375, Jan. 30, 1911. G. ORNSTEIN, 64 Kurfurstendamm, Berlin, W. 15, Ger. Mfg. acetylene tetrachloride by allowing CH: CH to act on Cl dissolved in CHCl<sub>2</sub>.-CHCl<sub>2</sub> in the presence of chem. active rays or of an agent capable of accelerating the chem. reaction, and insol. in CHCl<sub>2</sub>.CHCl<sub>3</sub> and non-vol.

Brit., 3,980, Feb. 16, 1911 CHEM. FABRIK GREISHEIM ELEKTRON, Frankfurt a/M, Ger. Mfg. phenylglycocoll salts by treating materials, obtained in the known manner from approximately 3 or more mol. proportions of aniline and 1 mol. proportion of chloroacetic acid and contg. phenylglycocoll anilide besides phenylglycocollaniline salt with approximately 1 equiv. of a neutralizing agent calc. for 1 mol. proportion of chloroacetic acid used in the process, sepg. the mixt. of phenylglycocoll aniline salt and phenylglycocoll anilide from the metal chloride, and transforming the mixt. thus freed from metal chloride into phenylglycocoll alkali salt by means of caustic alkali lye.

## 18. ACIDS, ALKALIES, SALTS AND SUNDRIES.

### T. LYNTON BRIGGS.

Retrospect and Outlook in the Realm of Applied Chemistry. OTTO N. WITT. Chem. Ind., 34, 89-94.—Optimistic. E. J. CRANE.

Conference on the Education and Training of Engineers, 1911. Electrician, 67, 543, 583.—A collection of papers by various English authorities. L. K. MULLER.

Relation between Practical Training and College Education for Engineers. H. Louis. Electrician, 67, 622. L. K. Muller.

The Debt of the Manufacturer to the Chemist. HARVEY J. SKINNER. Chem. Eng., 14, 307-8.

J. J. M.

The Consumption of the Commoner Acids in the United States. C. E. MONROE. J. Wash. Acad. Sci., 1, 70-1.

J. J. M.

History of Chemical Industries in Austria. PAUL MARTELL. Chem. Ind., 34, 205-10. E. J. CRANE.

Report on the Progress in Inorganic Chemical Industries. V. HOLBLING. Chem. Ind., 34, 398-402. E. J. CRANE.

Superphosphate in France. G. A. Br. Chem. Ind., 34, 96-7.—Curves showing the rise in 1906-7 and the fall in 1908-9-10 of the com. value of superphosphate in France as compared to that of the surrounding European countries. E. J. CRANE.

The Nitrogen on the World Market. G. A. Br. Chem. Ind., 34, 148. E. J. C.

Fifty Years of the German Potash Industry. P. KRISCHE. Gross-Lichterfelde. Chem. Ind., 34, 173-82.—A history of the chem. utilization of K salts in Germany from the beginning.

E. J. Crane.

The Italian Chemical Industry. D. G. Morselli. Chem. Trade J., 49, 150.
J. J. M.

Report of the Committee on the Testing of Chemical Reagents. L. F. KEBLER. Bur. Chem., Bull. 137, 50-1.—It is recommended that the designation c. P. be applied only to such chem. reagents as are free from recognizable impurities, that the term reagent be applied to all commonly employed chem. reagents which are free from all impurities to such an extent as to permit their use in all ordinary qual. and quant. analyses, that a sp. set of tests with which the chem. must comply be drawn up and adopted for each reagent, and that the term special reagent be employed only for certain reagents to be used chiefly for making special detns. which require absolute freedom from certain impurities.

H. C. FULLER.

Imports and Exports of Raw Materials and the Products of the Chemical Industries for 1910 thru the German Customs Department. Chem. Ind., 34, 418-24.—Statistics for 1910 compared to those for 1909.

E. J. Crane.

German Foreign Trade in Chemical and Pharmaceutical Products, Dyes and Paints According to the Quantity and Value in 1909 and 1910. Chem. Ind., 34, 101-10.—Import and export statistics.

E. J. Crane.

Imports and Exports of Raw Materials and the Products of Chemical Industries thru the German Customs Department for the First Quarter of 1911. Chem. Ind., 34, 256-64.—Quantity statistics.

E. J. Crane.

England's Foreign Trade in Chemicals in 1910. Chem. Ind., 34, 159-60.—Quantity and value statistics of imports and exports.

E. J. Crane.

Preliminary Report on the Importation of Chemical and Pharmaceutical Products and Raw Materials into the United States in the Fiscal Year Ending June 30, 1910. Chem. Ind., 34, 347-9.—Value statistics.

E. J. Crane.

Regeneration of Sludge Acid (WISPER). 22.

BLOUNT, B. AND BLOXAM, A.: Chemistry for Engineers and Manufacturers. Vol. 2. Chemistry of Manufacturing Processes. London: C. Griffin. 8°, 530 pp., 16 s.

DAMMER, O.: Chemische Technologie der Neuzeit. 2 Bd., Stuttgart: F. Enke. 35 M.

Directory of Chemical Industries throughout the World. Berlin: 727 pp., \$3.75. FISCHER, H.: Mischen, Rühren, Kneten und die dazu verwendeten Maschinen. Leipzig: O. Spamer. 90 pp., 7 M.

HAUSBRAND, E.: Das Trocknen mit Luft und Dampf. 4 Aufi. Berlin: J. Springer. 8°, 142 pp., 5 M.

JEEP, W.: Die Kitte und Klebstoffe. 5 Aufl. Leipzig: B. F. Voigt. 136 pp., 5 M.

NAGEL, O.: Transportation of Gases, Liquids and Solids. London: Vacher & Sons. 200 pp., \$2.50.

WICKOP, L.: Die Herstellung der Alkali-bichromate. Halle a/S, 6 M.

Workshop Receipts. 4 Vols. New York: Spon & Chamberlain. \$6.00.

Brit., 5,657, Mar. 7, 1910. J. PRICE, 17 Devonshire Square, London E. C. Mfg. ammonium nitrate by subjecting air or gases constituting air, H<sub>2</sub>O in a gaseous state or the gases constituting H<sub>2</sub>O, and additional supplies of O in combining proportions,

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to great heat and then suddenly cooling and liquefying them, whereby said gases combine to form NH, NO<sub>2</sub>.

Brit., 6,273, Mar. 12, 1910. J. W. BAILEY, E. 5th and Ingram Sts., Bayonne, N. J. Mfg. lead oxide by subjecting the particles of granulated or comminuted Pb in the presence of H<sub>2</sub>O to repeatedly recurring violent impacts and fractional attrition while suspended in or showered in or thru a body of air.

Brit., 10,522, May 1, 1911. FARBWERKE VORM. M. L. & B., Höchst a/M, Ger. Mfg. pure nitric oxide (NO) by subjecting HNO<sub>3</sub> contg. N peroxide, to electrolysis at the cathode.

Brit., 13,415, June 2, 1910. THE SALT UNION LTD., Colonial House, Liverpool. In the manuf. of salt from natural brine by the vacuum process of evapn., electrolyzing a sufficient quantity of brine to form NaOH equiv. to the Mg and Ca salts present in the mass of brine to be treated, partially carbonating the NaOH and then mixing the electrolyzed brine with the raw brine.

Brit., 13,506, June 3, 1910. T. F. Settz, Bahnstrasse 26, Kreuznach a/Nalse, Ger. A filtering sheet or plate consisting of a single layer or a plurality of superimposed layers of filtering material whose permeability decreases in the direction of flow of the liquid passing therethru.

Brit., 16,693, July 13, 1910. J. HARGER, Grange Hollies, Gateacre, near Liverpool. In the manuf. of oxygen-yielding mixtures contg. KClO<sub>2</sub> or other highly oxygenized salt mixed with (as a catalytic agent) Mn or Fe dioxide or other like peroxide, the use of the constituents in a state of very fine subdivision, with thoro mixing, the mixts. being compressed under very high pressure into a suitable form with 1 or more rods, wires, or the like passing therethru.

Brit., 20,414, Sept. 1, 1910. N. A. DEMONGEOT, 15 Ave. de la Bourdonnais, Paris. The use of crystallized CaCO<sub>2</sub> (either in the natural state as marble, Iceland spar, or the like, or as artificially produced in industrial chem. processes) as a support in fusing materials which are either vitrifiable or previously vitrified and obtained by any means in a state of fine division (in powder, granules, paste, or the like) and which contain sufficient fusible material (K<sub>2</sub>CO<sub>3</sub> or Na<sub>2</sub>CO<sub>4</sub>, KNO<sub>3</sub>, or the like) to enable the materials to amalgamate and form an integral body at a temp. below the critical temp. of decomp. of crystallized CaCO<sub>3</sub>.

Brit., 21,563, Sept. 16, 1910. BESSLER WARCHTER & Co., LTD., Salisbury House, Finsbury Circus, City of London. Mfg. a ferrated and borated alkali silicate by combining ferric oxide, calcined borax, and alkali silicate, or a mixt. of alkali silicates.

Brit., 23,148, Oct. 6, 1910. Wm. Anderson, John St. Factory, Govan, Scotland. Obtaining extracts from vegetable substances by bringing into contact with said substances the exhaust vapor from a turbine driving a centrifugal machine, allowing the vapor to condense thereon and dissolving the sol. matters, and simultaneously withdrawing the soln. by centrifugal action of the said machine.

Brit., 24,641, Oct. 24, 1910. A. MULLER, 5 H Dorotheenplatz, Leipzig, Ger. In the prep. of oxygen baths, combining the catalyzer with solid and little permeable bodies and bringing the same into contact with the bath H<sub>2</sub>O.

Brit., 25,400, Nov. 1, 1910. H. TIMPE, Weesperzyde 125, Amsterdam, Holland. Mfg. a plastic substance from a carbohydrate and an alkali albuminate by kneading a mixt. of the albuminate and a carbohydrate like starch or cellulose hydrate dissolved in alkali until it becomes a firm tough mass, then drying this mass, and finally pressing the mass in molds under great pressure.

Brit., 27,463, Nov. 25, 1910. R. P. PICTET, 116/7 Uhlandstrasse, Wilmersdorf,

Berlin. In the separation of oxygen and nitrogen in atm. air by passing air thru descending liquid N in a sepg. column, effecting the vaporization of the successively differing liquid mixts. by the liquefaction of N in a sep. coil passing thru the mixts.

Brit., 1,628, Jan. 21, 1911. P. SCHOU, 14 Gammel Mont, Copenhagen, Denmark. An app. for absorbing gases or vapors, comprizing a member of great surface area, adapted to be immersed in and withdrawn from the liquid, thereby subjecting the gas to be absorbed to a great surface or film of absorbing liquid.

Brit., 5,553, Mar. 6, 1911. CARL R. GUNTHIER, Meinersdorf, Sachsen, Ger. In a grinding and polishing body, the operative surface consists of artificial silk.

Brit., 5,577, Mar. 6, 1911. G. WANNSCHAFF, 44 Main Canal, Papenburg-on-the-Ems, Ger. Obtaining zinc oxide from materials contg. Zn or ZnO, by treating such material with a soln. of ZnCl<sub>2</sub> at a high temp., the soln. obtained contg. the ZnO and ZnCl<sub>2</sub> being cooled down and the ZnCl<sub>2</sub> soln. thereby recovered being again used for leaching, and the ppt. contg. ZnO and ZnCl<sub>2</sub> being extracted with H<sub>2</sub>O to recover the ZnCl<sub>2</sub>, whereby a further quantity of ZnCl<sub>2</sub> suitable for leaching is obtained, the solid residue consisting principally of ZnO with but small quantities of ZnCl<sub>2</sub> being capable of conversion into pure ZnO by alkalies or the like, such as milk of lime or soda lye.

Brit., 7,325, Mar. 23, 1911. P. R. ZIEROW, 179 Schonhauser Allee, Berlin, Ger. Mfg. a plastic substance by mixing crystallized pulverized gypsum or Iceland spar and MgO in the form of magnesia usta with H<sub>2</sub>O, then adding this mixt. to a soln. of gelatin in H<sub>2</sub>O and finally adding glycerol and wax or a wax-like substance with or without the addition of coloring matters, heat being applied during the operation.

Brit., 9,921, Apr. 24, 1911. LEON DEVOS, Hampden House, Kingsway, London, W. C. A powder preparation for making a solution for use in combination with an aluminum surface for cleaning silver, electroplate, jewelry, and the like, composed of a large proportion of anhydrous Na<sub>2</sub>CO<sub>2</sub> and a small proportion of anhydrous K<sub>2</sub>CO<sub>3</sub>.

## 10. GLASS AND CERAMICS.

### G. E. BARTON, A. V. BLEININGER.

Efflorescence on Brickwork. The Builder, 1911; thru Brit. Clayworker, 20; Brickbuilder, 1911, 42-3.—Special sources of scum are: (1) FeS<sub>2</sub> in clay, later oxidized to sulfate, (2) ashes used to prevent shrinkage, (3) high S in coal. Condensation of moisture during drying must be avoided. To remove scum from walls, wash alternately with hard soap (2.2 lbs. per gal.) and  $Al_2(SO_4)_2$  (1 lb. per gal.). CaSO<sub>4</sub> scum may be removed by 1:5 HCl.

C. H. Kerr.

A Pyrometer for Hoffmann Kilns. Anon. Brit. Clayworker, 20, 82.—An adaptation of the new Féry spiral radiation pyrometer. A cap is fitted to the head of the pyrometer so that the instrument can be placed directly over the fire hole. By brushing dust around the joint, all cold air can be excluded.

C. H. Kerr.

Burning in the Buhrer Fan Kiln. J. Graham Maxwell. Brit. Clayworker, 20, 81.—British clays will not stand the rapid burning of the fan kiln. The cost of running the fan is high. An efficient stack will produce all the draft required and at a lower cost.

C. H. Kerr.

Lead Poisoning. EDIT. Brit. Clayworker, 20, 72-3.—Ann. Rept. of Chief Inspector of Factories and Workshops. Since 1899 the no. of cases has decreased greatly. In 1910 there were 77 cases in England. Serious attempts are being made to use low solubility glazes; some now contain under 2% sol. Pb. One or two factories use lead-

less glazes successfully. The chief preventives are, (1) good ventilation, (2) reasonable temps. for working, (3) scrupulous cleanliness.

C. H. KERR.

A Course of Instruction for Foreman. EDIT. Brit. Clayworker, 20, 76.
C. H. KERR.

CHAPIN, H. C.: How to Enamel. New York: Wiley. 70 pp., \$1.00. Solon, M. L.: Ceramic Literature. Lippincott. 649 pp., \$15.00.

Brit., 14,981, June 22, 1910. K. A. MANKAU, 7 Mytninskara, St. Petersburg, Russia. Firebricks and like refractory articles with a small % of spinel as a cementitious or binding material.

### 20. CEMENT AND OTHER BUILDING MATERIALS.

C. N. WILEY.

Ferrite Cements and Ferro-Portlands. E. C. ECKEL. Eng. News, 66, 157-8. The author divides Portland cements and their allied products into these classes: (A) high-Al cements,  $(A_1)$  white Portlands whose Al<sub>2</sub>O<sub>2</sub> to Fe<sub>2</sub>O<sub>2</sub> ratio varies from 6: 1 to near infinity, (A2) normal Portlands, Al2O2 to Fe2O2, ratio 1: 1 to 6: 1 usually between 2: 1 and 3: 1 and (B) high-Fe cements, (B<sub>1</sub>) ferro-Portlands, Fe<sub>2</sub>O<sub>2</sub> to Al<sub>2</sub>O<sub>3</sub> ratio ranging from 1: 1 to 4: 1, (B<sub>2</sub>) ferrite cements containing little Al<sub>2</sub>O<sub>2</sub> and a Fe<sub>2</sub>O<sub>2</sub>. Al<sub>2</sub>O<sub>2</sub> ratio above 4: 1. The author proposes to manuf. this "ferro-cement" using as the principle raw material glauconite or green sand, the comp. of which in % is approx. SiO, 50, FeO 30, KaO 8 and AlaO, 3-8. To this must be added lime. A cement made by the author, using a limestone rather high in Mg, (anal. in %: SiO<sub>2</sub> 19.52, Al<sub>2</sub>O<sub>3</sub> 3.30, Fe<sub>2</sub>O<sub>3</sub> 7.38, CaO 62.62, MgO 4.12, SO<sub>3</sub> 0.42, loss 1.48) had these physical properties in fresh and salt water resp.: initial set 6 hr. 5 min., 5 hr. 30 min.; final set 9 hr. 10 min., 9 hr. 5 min.; tensil strength 24 hr. neat, 267, 368; 7 days' neat, 688, 881; 7 days 1:3 sand, 195, 285; 28 days' neat, 888, 887; 28 days 1:3 sand, 322, 321. This green sand occurs in large deposits in New Jersey and can be placed aboard cars at from 15-30c per ton. Using green sand the cost of manuf. per bbl. would be about the same as for Portland, using Fe-ore the cost is about 20c per bbl. higher.

C. N. WILEY.

Cement Manufacture in Germany. Otto Schott. Mining Sci Press, 103, 194.—Many works of North Germany make their cement from chalk and a clay containing much sand and flint, and since these contain a high % of moisture the wet process is always given the preference. Recently 7 works have started to prepare hard materials like limestone in the wet way by the so-called thick slurry process but the dry process is considered the better and more economical by most German cement makers.

E. J. Crane.

Testing of Portland Cement. A. C. Davis. Concrete Constr. Eng., 6, 418-29.

—A Cambridge Univ. lecture discussing cement in terms of Brit. Stan. Specifications.

A. J. Phillips.

The Rise in Temperature during Setting of Concrete. J. G. LEIGH. Concrete Constr. Eng., 6, 416-7; Ciment, 16, 103-4.—At several points in the locks of the Gatun Dam at Panama, thermocouples were inserted. Time-temp. curves show that the max. rise occurred in 8-15 days and the temp. depended on the position of the thermocouple in the concrete.

A. J. PHILLIPS.

Testing Clinker Concrete. J. C. DAVIS. Munic. J. Eng., 36, 176-7.—Tests of

concrete beams made from clinker, of a garbage incinerator—resulting from the burning of a mixt. of garbage refuse and ashes—showed that it is stronger than gravel concrete, far superior to cinder concrete, may be used in mass concrete, but tests have not been carried far enough to det. its value for reinforced concrete. A. J. PHILLIPS.

Blast Furnace Slag and Concrete. Anon. Stahl u. Eisen, 31, 1154-5.—A review of the work of Schick (Zement und Beton., 1910, 782) on Thomas slag and of Dahlmann (Ibid., 1911, 262) concluding that slag concrete is equal if not superior to sand concrete on account of the presence of material varying from large lumps to fine sand.

A. J. Phillips.

Reinforced Concrete Specifications. Anon. Mining Sci. Press, 103, 141.—The Science Comm. of the Concrete Inst. investigated the rusting of steel in concrete and were led to conclude that: The sand, stone and cement must be thoroly mixed, properly proportioned, fairly wet, and well hammered into place to minimize voids. The aggregate should pass a 3/4-in. sieve and be as non-porous as possible. The concrete covering should never be less than 1/4 in. thick, and in the case of round bars, as thick as the diam. of the bar. In structures exposed to  $H_2O$  or damp air, the thickness should be increased at least 50%, and the size of the aggregate reduced to obtain a dense skin. Sufficient space should be allowed between each piece of reinforcement and its neighbor so that each piece may be completely surrounded by concrete. All steel, should be firmly supported during the ramming to avoid displacement. Thick rust should be removed by scraping but the reinforcement must be neither oiled nor painted.

E. J. Crane.

Proposed Provisional Tests for Road Materials. L. W. PAGE. Eng. Contracting, 36, 125-6.—A comm. report on standard tests for road mat. to the Am. Soc. Test. Materials. Determination of soluble bitumen: If hard and brittle the material is ground, spread in an open Ni dish and kept at 125° for 1 hr. In case it is not ground heat in an oven, then crush and dry. Tar or oils may be dehydrated by distillation, the water-free distillate being returned to original residue. Weigh 2-15 g. of dry mat. (insuring presence of 1-2 g. pure bitumen) into a 150 cc. tared Erlenmeyer flask, treat with 100 cc. CS, shake and let settle 48 hrs. Decant the soln. into a similar weighed flask without disturbing the residue and again treat contents of first flask with CS, and let settle 48 hrs. Decant the liquid (kept at 25°) in the 2nd flask thru a weighed Gooch crucible, the filter being of ignited amphibole (long fiber) not over 1 in. deep. Wash the residue on the filter and in both flasks with fresh CS, and allowed to settle well (24 hrs.). Filter the solns. in both flasks and wash till colorless, dry the crucible at 125° and weigh. Evap. the filtrate, burn the residue and add the wt. of ash to the wt. of residue in the crucible. The wt. of sample minus this sum gives the sol. bitumen. Determination of penetration of bitumen: The penetration shall be the distance expressed in 0.01 cm. that a no. 2 needle will penetrate vertically without friction at 25° (sometimes also at 0° and 46°) under a stated wt. for a stated length of time; the time and wt. factors being detd. as follows: First test for 5 sec. with 100 g. wt.; if the result is less than 10, try 200 g. for 1 min. If between 10 and 300, penetration is detd. under 100 g. for 5 sec.; if greater than 300, 50 g. for 5 sec. is used. When testing material softer than 100 penetration a container not less than 11/4 in. in diam. is used. Determination of loss on heating: In a cylindrical tin box 2 cm. high by 6 cm. in diam. place 20 g. of water-free sample Penetration is detd. at 25°. Heat in a hot air oven (New York Test. Lab. Oven without fan) to 163° for 5 hrs. Cool, weigh and det. penetration at 25° and find loss in penetration. Sizing and separating aggregate in asphalt paving mixtures: Pass sample thru 200, 100, 80, 50, 40, 30, 20, A. J. PHILLIPS. 10 meshes per lin. in. screens in the order named.

The Preservation of Structural Timbers from Decay. C. P. Winslow. U. S.

Forest Products Lab. Proc. Eng. Soc. Western Penn., 26, 427.-Modern methods of preservation depend upon impregnation with materials poisonous to the fungi. Creosote oil does not depend for its preservative action upon its content of tar acids and naphthalene as much as upon the anthracene fractions, since these are more lasting. Since American coal tars are distilled for the production of soft pitch rather than for creosote oil, the distillation is not carried as far as it is abroad where the anthracene oils are desired for dye manuf. The imported creosote oils therefore are more in demand. Crude oil owes its preservative properties to its power to plug the pores and prevent admission of air and moisture. ZnCl, is efficient but leaches out with water. Creosote oil is the more effective but costs 4-10 cents per cu. It. of timber, while ZnCl, costs 2 cents or less. The processes of impregnating timber are described, the economic aspects of wood preservation discussed, and specifications for coal-tar creosote as adopted by the Railway Maint. of Way Assoc. and as proposed by the U. S. Forest Service given. In the discussion many commercial processes are described and illustrated. HORACE C. PORTER.

Methods and Apparatus for the Examination of Coal Tar Used in Road Work.

J. HUTCHINSON. Eng. Contract, 35, 711.—Directions for distillation, sp. gr. free C and evapn. tests simplified for road engineers.

A. J. PHILLIPS.

The Prevention of Corrosion in Metal Laths. C. W. Nobel. Cement, 12, 48-53.—A study of protective coatings for metal laths was made, in order to det. which coating would best withstand plaster of Paris, and which sets up elec. currents and thereby causes the O mols. to attack steel. Ingot iron for laths was rejected, because a chem. analysis would be required to guarantee the purity of the Fe. All paint, japan and asphalt varnish coatings were rejected as being either so soft as to scratch readily, thus allowing penetration of moisture, or so brittle as to be unable to withstand wear and tear in handling. Hot and electro-galvanized Fe are too expensive. Zn is attacked by plaster and attempts to substitute Pb or a Pb-Zn alloy failed, as Pb is electro-negative to Fe. Sherardized coatings were finally adopted since there is no contact between 2 metals of apparent variation in elec. potential but a gradual passage from pure Zn thru Fe-Zn alloy to pure Fe.

A. J. Phillips.

Efflorescence on Brickwork. 19.
Flushing Streets. 14.
Good Roads (ALEXANDER). 22.

Brit., 30,035, Dec. 24, 1910. E. E. HIPPE, 31 Mariendalsvej, Copenhagen, Denmark. Mfg. artificial stone such as marble, granit, malachite, serpentine, Labrador stone, and the like, by molding a mass of hydraulic or other cement or material, by first producing in a mold or upon a suitable foundation a film of a soln. of a salt of a heavy metal other than a salt of an earthy alk. metal, uncolored or colored or having a coloring effect because of its reaction or of added coloring material, and in some cases having an addition of a material for retarding the setting, so that it forms a thin or semi-liquid cement, and then putting upon this film 1 or more colored or uncolored cements or molding materials, which because they contain alk. or earthy alk. compds. react upon the film or metal salt, while forming insol. compds which render the superficial structure of the molded articles harder and denser and in some cases aid in the production of special shades of color.

Brit., 5,079, Mar. 1, 1911. J. J. Fraser, 13 Carnarvon St., Glasgow. Mfg. artificial stone from a mixt. of ground or crushed slag, coal sludge, calcined magnesite, and MgCl, with or without the addition of calcined MgO, and sand or oil or coloring, or all of these.

### 21. FUELS, GAS AND COKE.

#### J. D. PENNOCK.

The Purchase of Fuel upon a Heat Unit Basis in Germany and Switzerland. J. B. C. Kershaw. Met. Chem. Eng., 9, 427.—In Hamburg, the Smoke Abatement Soc., which has over 1300 boilers or other furnaces under its control, has arranged to undertake the regular sampling of the fuel supply for its members, which is tested by a fuel expert for calorific value, ash and moisture on the trial samples and for ash and moisture on the subsequent samples on the same contract and the calorific values calculated from these. In Switzerland the State tests all fuel for railroads and gas making.

E. C. S.

Plant of the Bailey Wood-Coal Company. H. D. JACKSON. Eng. Mining J., 92, 319-20.—Description of a West Virginia power plant of up-to-date design.

ROBERT KANN.

Vertical Retort Tar as Fuel for Diesel Engins. W. ALLNER. Progressive Age, 29, 481; J. Gasbel., Apr. 8, 1911; J. Gas Lighting, 115, 106.—In testing a 100 h. p. Diesel engin of Körting Co., good results were obtained when an auxiliary fuel in the shape of paraffin oil was used to ensure ignition. Tar fuel is the least expensive for Diesel engins and with slight changes in the engin, no difficulty is experienced in using it.

E. C. S.

Titration Method for Determining Sulfur in Coal. E. A. CUNNINGHAM. Chem. Eng., 14, 287.—This method was devized for detg. S in coal from the washings of the Atwater Mahler bomb calorimeter and is as follows: To the bomb washings, which have been titrated as usual to det. the calories, add exactly 9 cc. of a 0.5 N BaCl, soln. (equiv. to 7.2% S on 1 g. coal), then 5 or 6 cc. N Na<sub>2</sub>CO<sub>3</sub> soln., filter and wash with hot water and return filter and contents to flask, add about 15 cc. water, cork and shake violently to break up paper; wash off cork and down sides, add Me orange and titrate the BaCO<sub>4</sub> (due to excess of BaCl<sub>2</sub>) with 0.0625 N HCl, subtracting cc. found from cc. given in a blank with same amt. of BaCl<sub>2</sub>. The HCl soln. = 0.001 g. S per cc. The solns. are standardized by using a soln. of Na<sub>2</sub>SO<sub>4</sub>, the S content of which has been carefully detd. gravimetrically. The bomb washings need not be filtered before adding the solns. The BaCO<sub>3</sub> with the BaSO<sub>4</sub> entirely prevents the tendency of the latter of running thru the filter. About 10 min. is required. Results reported show excellent agreement with the gravimetric method.

F. W. Smither.

Determination of Phosphorus and Sulfur in Coal and Coke. W. I. KEHLER. Metal Ind., 9, 342-3.—Phosphorus: Transfer the ash from 2 g. coal or coke, ground to pass a 100-mesh sieve, to a 300 cc. Erlenmeyer flask, add 20 cc. conc. HNO, and 5 cc. conc. HCl, evap. to dryness and bake for 5 mins. after the cessation of all fumes. Take up in 15 cc. conc. HCl, again evap. to 5 cc., dil. and filter from the SiO<sub>2</sub>. Next evap. the soln., to which 20 cc. conc. HNO, have been added, down to 5 cc. again, add 20 cc. of a satd, soln. of chromic acid in conc. HNO, and continue the evapn. to about 15 cc. Ppt. the P by adding 50 cc. molybdate soln. (as per Blair) heated to 60°, and finish as usual in Fe or steel. Sulfur: 1 g. coke or hard coal and 0.5 g. soft coal, ground to 100 mesh, is intimately mixed in a 20 cc. Pt crucible 3/4 full of the usual Eschka mixt. Heat under constant stirring (about 10 min:) until the gray color has given way to a yellow or red-brown and all the C has been consumed. Cool, add r g. dried and powdered NH, NO, and heat (covered) at a low red heat for 2 min. with a slow flame. Then uncover and ignite strongly for another minute. Boil 2 min. in a beaker, filter, wash with hot H<sub>2</sub>O, add to the filtrate 15 cc. of conc. HCl, 8 cc. BaCl<sub>2</sub> (20%) and sufficient hot H<sub>2</sub>O to bring the vol. to 100 cc., filter off BaSO<sub>4</sub> and weigh. ROBERT KANN.

Gasoline from Natural Gas. Anon. Mining Sci., 64, 55.—The first effort to make practical use of the natural gas which is going to waste daily in the oil fields east of Boulder, Colo., is now under way with the completion of a gas-compressing plant. Its capacity is 300,000 cu. ft. of gas daily. The yield is 3.5-5.5 gals. per 1000 cu. ft. of gas.

R. E. H.

Calculation of Combustion Gas Analyses. R. Jeller. Z. anal. Chem., 50, 250-63.—In connection with a recent article on this subject by G. de Voldere and G. de Smet (C. A., 5, 1041, 2423) the author brings out some observations of a similar nature made by him some years ago. The discussion is largely of an algebraic nature. By an appropriate combination of combustions and absorptions, a sufficient number of condition equations could be obtained for detg. all the constituents of a gas, provided that its qual. comp. is known.

G. N. T.

Lignite as a Source for Gas Supply. Anon. Braunkohle, Mar. 17, 1911; thru Progressive Age, 29, 482.—The use of lignite for generation of gas for power purposes is discussed. Suction gas plants are especially suitable. The gas has about the same calorific value as coke producer gas. Modern plants operate on about 1.6 lbs. coal per h. p. hr.

E. C. S.

Purification [of Gas]. A. B. LITTLE. Am. Gas Light J., 95, 5-7, 17-9.—The author gives an extended résumé and discussion of the subject and suggests several points for investigation.

E. C. S.

Long Distance Gas Distribution, Its Application and Economy. G. B. E. Blum. J. Gas Lighting, 115, 102-5.—An address. C. A. C.

Pyrometer Installation at Cambridge Gas Works. Anon. J. Gas Lighting, 115, 162-3.—An Fe runner is fixed overhead parallel to and in front of the retort beds. The runner supports a carriage from which is suspended a long vertical rod. On this rod is a Féry telescope which may be adjusted to any of the retort doors. From the telescope and small thermocouple, the e. m. f. set up is conducted to a millivoltmeter at one end of the retort house, where the temp. may be read off from any retort.

C. A. C.

Modern Gas Producers. J. Hofman. Iron Age, 86, 280-1.—Abstract; see C. A., 5, 1897.

L. A. TOUZALIN.

Recent Advances made in the Manufacture of Incandescent Gas Mantles. R. C. Boehm. Illum. Eng., 4, 461. C. G. F.

The Work of Engineers in the Gas Industry. F. P. ROYCE. Chem. Eng., 14, 319.

J. J. M.

The Destruction of Firebrick in Coke Ovens and the Reasons Therefor. B. Kosmann. Stahl u. Eisen., 31, 730-1.—The cracking of the firebrick lining of coke ovens is ascribed to the entrance of particles of C into the red-hot lining, which, possibly thru the intermediate formation of Si carbides, are finally deposited in the pores as graphite. Note. F. Schreiber. Ibid., 731-2.—Objects to K's conclusions and thinks the destructive action largely due to the penetration of the firebrick by salts, with the formation of less resistant and more volatil compds. M. Heidelberger.

Automatic Gas Analysis. 1.

Coal-like Masses of Compositae (DAFERT, MIKLANZ). 11.

Distillation of Sewage Sludge (BAYER, FABRE). 14.

Detection of Inflammable Gas (PHILIP, STEEL). 1.

Examination of Coal Tar (HUTCHINSON). 20.

Sulfur in Tar Residues (HUBBARD, REEVE). 22.

Vegetation of the Carboniferous Period (DACHNOWSKI). 8.

GISSING, F. F.: Commercial Peat: Its Uses and Possibilities. Lippincott. 200 pp., \$2.00.

HOLMES, J. A.: Sampling of the Coal in the Mine. Bureau of Mines.

LEEDS AND BUTTERFIELD: Acetylene: The Principles of its Generation and Use. Second edition. J. B. Lippincott. 411 pp., \$2.75.

STRACHE, H.: Jahrbuch über die Fortschritte in der Gasbeleuchtung und Gasindustrie 1910. München: R. Oldenbourg. 4.80 M.

Brit., 6,041, Mar. 10, 1910. N. TESTRUP, 6 Broad St. Place, London Co. Wet carbonizing peat by passing it thru an app. adapted for the regeneration of heat imparted to and produced from the peat, and allowing continuous flow of peat thru it, without ebullition of the heated material, the peat being raised to 180° or thereabouts, to obtain the automatic rize of temp. which then results by reaction within the mass, and in which the process is then allowed to be completed, wholly or in part, by the internally generated heat.

Brit., 6,899, Mar. 18, 1910. K. HUESSENER, 38 Upper Thames St., London, B. C. In the separation of tar from hot dry distillation gases by means of tar or tar and gas H<sub>2</sub>O, the tar or tar and gas H<sub>2</sub>O is introduced under pressure into the hot distillation gases in a part of the delivery end of the ascension pipe, and the hydraulic main.

Brit., 10,832, May 2, 1910. A. B. HILL, Chicago, Ill. A fuel consisting of finely divided coal 1 ton, maix oil obtained from crude petroleum by distilling off the light ends 20 lbs., black rosin 12 lbs., and lime  $H_2O$ .

Brit., 10,841, May 3, 1910. E. CAVERNIER, 173 rue de la Republique, Puteaux, Seine, France. In the manuf. of fuel using cellulide as agglutinant, the fuel after being molded into briquets is heated in an oven having several chambers in which the briquets are heated and afterwards cooled in an atm. free from O.

Brit., 12,613, May 24, 1910. E. TRAINER. Block fuel briquets. See C. A., 5, 190.

Brit., 15,817, July 1, 1910. M. G. CHRISTIE, near Wakefield. In the process of recovering ammonia from moist gases by passing them thru a saturator maintained at a temp. high enough to prevent condensation of the moisture from the gas, passing SO, into the liquor in the saturator, or into liquor contd. in a sep. bath and circulated thru the saturator, and at the same time electrolyzing the liquor.

Brit., 15,877, July 2, 1910. K. BURKHEISER, 12 Forster Strasse, Aachen, Prussia. In the prep. of ammonium sulfite or ammonium sulfate from gases, products of combustion of distillation gas from coke ovens and the like contg. H<sub>2</sub>SO<sub>4</sub> are brought into contact with washing liquor previously rendered alk. by the absorption of NH<sub>2</sub> from the said distillation gas, the said alk. liquor absorbing SO<sub>2</sub> and the same liquor being then again conducted into contact with the said distillation gas contg. NH<sub>2</sub> to be again rendered alk., and after depositing the salt, again being brought into contact with the said products of combustion, the constantly circulting liquor thus forming, at different stages in its circulation, an agent for the reaction of the 2 bodies given up to it, and obviating the mixt. of the said gases of distillation and combustion, the sulfate or sulfite of NH<sub>2</sub> formed being continuously removed from the washing liquid.

Brit., 19,008, Aug. 12, 1910. H. BREITBART, 308 Kaiserstrasse, Duisburg-Beeck, Ger. For ascertaining the proportion of ignitable admixture in the air by rendering the latter explosive by the introduction of a predetd, proportion of ignitable substances, with or without compression, the added ignitable substances consisting of an easily vaporizing liquid fuel.

Brit., 22,834, Oct. 3, 1910. A. A. A. ZIMMER, 29 Hartham Road, Holloway, London. Mfg. fuel briquets by adding coal dust to a mixt. of benzene, acetone, nitrocellulose, and pitch, then partially or wholly evapg. the solvent before forming the briquets and finally pressing and drying the formed briquets.

Brit., 5,667, Mar. 7, 1911. R. FABRY, The White Bldg., Titzalen Square, Sheffield. In the recovery of ammonium sulfate direct from coke oven or retort gas of the kind described in 4,473, of 1910, the ZnS produced is made to react with H<sub>2</sub>SO<sub>4</sub> so as to form ZnSO<sub>4</sub> and H<sub>2</sub>S, the former either in crystals or in soln.

## 22. PETROLEUM, ASPHALT, COAL TAR AND WOOD PRODUCTS.

#### R. E. HUMPHREYS.

Optical Activity of Petroleum. J. BOBCZYNSKI. Lemberg. Petroleum, 6, 438-9.

—The exam. of the optical activity of several crude oils from Galicia and Patagonia showed that a gradual rearrangement takes place in the oils, and indicates that all oils must, after a definit period, lose their activity, notwithstanding the stability of their active constituents. The influence of the mass of the radicals and the nature of the elements in them, the various kinds of isomerism, the compds. in the oil, the ring systems, the temp. and conc. coeffs, and the nature of the solvent, upon the optical activity of the oils are discussed. The similarity in optical relations of the cholesterol derivs. and petroleum distillates is pointed out. No ozonides could be obtained on treating the higher-boiling distillates of petroleum and cholesterol with ozone. By means of the Lippich polarimeter, the influence of color upon the activity was studied. The differences in rotation were found to be significant.

O. E. Bransky.

The Expansion Coefficients of Roumanian Oils and their Derivatives. C. T. Petrroni. Rev. génerale, 5, 21; thru Petroleum, 6, 607.—The coeffs of 16 oils, 20 fractions from Moreni and Campina crude oils, 12 lamp oils, 3 benzines, and 14 lubricating oils and residues were detd. Tables are given, and the relations between these constants and the sp. gr. and flash pt. are discussed. For the correction of sp. gr. from t' to t, knowing  $\alpha$  (expansion coeff.) the following formulas are used: When t' is higher than t,  $\beta = [Dt(t-t')\alpha]/[1+\alpha(t-t')]$ . When t' is lower than t,  $\beta = [Dt(t-t')\alpha]/[1-\alpha(t-t')]$ . O. E. Bransky.

The Expansion Coefficients of Roumanian Oils and their Derivatives. C. CONDERA. Petroleum, 6, 608.—The constants were obtained by means of the Holde dilatometer. Large variations were found in the heavier distillates, and, because of their uniformity of comp., only small differences in the benzines. The following corrections are given: sp. gr., 0.600-0.670, correction, 0.001 for 1°; 0.670-0.750, 0.0009 for 1°; 0.750-0.820, 0.0008; 0.820-0.890, 0.0007; 0.890-0.990, 0.0006. These corrections are applied to the hydrometer readings.

O. E. Bransky.

Lubricating Oils and their Forms of Application. R. Adan. Gent. Lab. *Echo Minier Ind.*, 1911, Feb.; *Anal. ind. electrochim.*, Sept.; thru *Chem. Zentr.*, 1911, I, 1898.—A compendium of the necessary requirements of lubricating oils for a variety of purposes, with tables of tests and the deductions to be made therefrom.

M. HEIDELBERGER.

Differentiation of Oils under Sections 177 and 178 of the Austrian Tariff Laws.

L. Singer. Petroleum, 6, 604-5.—Refined or semi-refined light oils of sp. gr. 880 or less are classed under 177, while refined and lubricating oils, containing vegetable oils and fats, with a sp. gr. above 0.880 included in section 178. Oils yielding on distillation 10% or more by vol. at 300° are regarded as illuminating oils and come under 177.

R. Kind and the writer consider the distillation test as impracticable; the former proposes that oils with a flash pt. under 140° be classed under 177. The writer claims that the flash pt. does not give a good classification. He points out that a gas oil, from Boryslaw crude oil, sp. gr. 0.868, showed a flash pt. of 134° in open cup, and 14% off at 300°, and yet was very unsuitable for a lamp oil. He suggests a viscosity test, in conjunction with the sp. gr., flash pt., and distillation tests, as a conclusive differentiation.

O. E. Bransky.

The Regeneration of Sludge Acid of Petroleum Refineries. P. WISPEK. Lemberg. Petroleum, 6, 1045-8.—Sludge acid is divided into 2 groups, the H<sub>0</sub>SO<sub>4</sub> sediment (1) from the refining of benzine and refined oil, (2) from the refining of lubricating oils. In the former, 10% of the acid is consumed, being decomp, or combined; 100 pts. by wt. of unused acid of 66° Bé. yield 125-130 pts. of 50-55° Bé., containing in soln. asphaltic and sulfurous org. matter. By dilg. this acid to 35° Be., most of the oily matter (20-25% by vol.) is sepd. The bottom layer consists of dil. H.SO4 which must be steam distilled and conc. Since this conc. acid is usually black, and contains undesirable org. compds., various methods have been devized for clarifying it. Diffusion thru a porous diaphragm against water gives a clear acid, but organic and sulfo acids are also carried along, and on evapg, the acid, the latter are decompd, and the acid turns black. Regeneration by means of Si compds. yields a yellowish acid, which becomes black when evapd. Filtration thru "spodium" and heating with HNO, failed. Evapn. in Pb pans to 60° Bé., and then in porcelain dishes to 66° Bé. is the process generally used, but much SO, is evolved, and the acids foam considerably. The best method is that of L. Balcher and S. Stenzel used by Steaua Romana Petroleum Co. at Campina, Roumania. This process yields a clear, transparent acid of 97-98% H-SO4, and amtg. to 75-80% of the original vol. of acid, 100 pts. by wt. give 130 pts. of sludge acid, containing 90 pts. of HrSO4; of these 90 pts., 75-80 pts. are recovered. The sludge acid i washed with 50% by vol. of water, and the oil removed; this oil, amtg. to 20-25 pts. by vol., is used as fuel for the distillation and corc. of the dil. acid. Before use it is washed 2-3 times more; wash water is used for dilg. the new sludge acid. The dil. acid (35-40° Bé., and 125-130 pts. by vol.) is evapd. in Pb pans to 60° Bé. and run into the distilling retorts, the bottom pans of which are made of cast iron and the upper sections of acid- and fire-proof brick (Volvic lava is best). The bottom is filled with pure, clear, conc. H<sub>2</sub>SO<sub>4</sub>, heated to its b. p., 300°, and air continually forced thru. The inflowing 60° Bé. acid is immediately vaporized. The asphaltic and other organic impurities are completely oxidized to CO<sub>2</sub>. The escaping gases consist chiefly of CO, and contain but traces of SO. The oxidation takes place at the expense of the air, and not of the acid. A pure, transparent inflowing acid of 60° Bé., distils over into the receiver. The amts. of distillate and inflowing acid must be kept equal, in order not to change the conc. of the acid in the still. The 60° Bé. is warmed in Pb pans, then allowed to flow into a second series of stills containing heated conc. acid. Dil. H.SO, of about 40° Bé. distils over; this acid is evapd., and then redistilled with the 60° acid. A pure, transparent acid of 66° Bé., containing 97-98% H<sub>2</sub>SO<sub>4</sub>, is drawn O. E. BRANSKY. off from the stills.

Oil Fuel for Steam Boilers. B. R. T. Collins. J. Am. Soc. Mech. Eng., 33, 930.—The advantages and disadvantages of oil fuel as compared to coal are discussed. Typical analyses of crude and fuel oil are given, the principles involved in efficient

oil burning are stated, and the most important types of oil burners illustrated and described. There is a small but steadily increasing field for the use of oil fuel for steam generation in the Atlantic Coast states.

HORACE C. PORTER,

The Oil Fields of Sakhalin. G. BEHAGHEL. Petroleum Rev., 24, 193-5, 225-7, 269-70.—Sakhalin, an island in the northern continuation of the Japanese Archipelago, has oil resources near the east coast. Boring has been going on for 18 mos. with considerable success. The geological structure of the island is remarkably similar to that of the Japanese island Hakkaidot. Two high ridges stretch parallel to each other in a meridional direct on. Strata of the upper Cretaceous age, enclosing coal seams, lean against the western sides, while on the east Tertiary formations predominate. Few eruptive rocks are to be found. Pressure, rectangular to the main ridges, has wrinkled the strata into folds parallel to the main ridges both on the eastern and western coasts. Within those on the east are the oil-bearing layers. This condit on does not conform to the old theory that oil in quantity can be found only in anticlinal portions of folded country. All the strata east of the main ridges dip toward the coast at an angle of 40-60°. There is here nothing to prove the existence of a true anticlinal fold with sides dipping away in different directions from a supposed axis. Folds have undoubtedly been formed in the strata of the east coast, for widely separated and parallel outcrops have been found identical in structure and fossils. This reduplication of strata is explained by a tipping over of the folds toward the west with subsequent erosion. The author believes the formation to be Pliocene. The coast rizes by terraces, and beyond the western boundary of the second terrace appear a series of swampy depressions, with oil shows, which comprizes the oil zone. Beyond this, other terraces extend to the eastern one of the 2 main crests. The oil zone is from 1/4 to 2 miles wide and 240 miles long. Nearly all the swamps are covered with oil, and large areas are covered with a dark-brown, tough mass called "kir," formed by evapn. and oxidation of the outcropping oil. This is similar to asphalt, and is thick and strong. There are many gas outcrops of all sizes. The oil and gas shows appear to be situated on a line of fissures and not on an outcropping of the oil-bearing strata. No borings have been made east of the outcrops, so it is not known if there are strata in the depth the outcrop of which would coincide with the oil shows; but borings within the zone have discovered oil strata for which no outcrops of petroliferous sandstone could be found further west. So the oil line marks deep fissures extending to deeper deposits: said fissures coinciding with a fault. The oil layers are in 2 groups, 500 and 700 feet in depth. They are probably not the Primary deposits but the result of infiltration, and deeper borings will undoubtedly open a great reservior. The quality of the oil is good, getting lighter and richer with increasing depth. Analysis gives 0.5% of benzine, 35-44% kerosene, and of a nature similar to that of Baku. Transportation and refining problems will be of easy solution. These oil fields are to be of extreme importance to Japan, Asiatic Russia and China. R. E. H.

Geological Structure and the Production of Oil Found South of St. Geran in Jelisawetpol. Y. Striyov. Mitt. Ges. Naturforscher, Kiev, 1910, 307-16; thru Petroleum, 6, 1061-2.—The oil is supposed to have curative properties especially in cases of rheumatism, burns, skin diseases, and is used in the prep. of 2 salves, "Naphthalan" and "Kojolan," which are widely used in the Orient. Many visit the place to take oil "baths." The oil is dark green, free from acids and paraffin, and leaves no residue on burning; it has sp. gr. 0.960, flash pt. 140°, viscosity 820 secs. at 50°, chill test—20°. By distillation, 35% light oils, 45% cylinder oils, and 20% pitch are obtained. The oils deposit seem to be of a secondary character, since a light, undecomp. oil (the mother oil) is found in lower strata.

The Government Earth Reservoirs for Petroleum at Boryslaw. P. WISPEK. Petro-

leum, 6, 2021-4.—Description of reservoirs erected by the Austrian gov. to store its crude oil. It is for the purpose of storing the crude oil, while the sepn. of the lighter constituents (benzine) is being made, that these earth reservoirs were constructed.

O. E. BRANSKY.

New Theories on the Formation of Petroleum. A. Gerbault. J. pétrole., 11, 71.—Rakuzin considers all petroleums to have optical properties characteristic of organic matter, especially their action on polarized light. Chardin thinks that petroleum is formed in the depths of the earth by the action of heat on H<sub>2</sub>O and rocks. It is forced up by the pressure of gas and comes in contact with animal and vegetable remains, when it partly dissolves these and acquires its definit comp. F. M. ROGERS.

Sampling Shipments of Oil. Anon. Eng. Mining J., 92, 393.—Directions published by the Bur. of Mines for taking samples, on which the calorific power and other consts. are detd.

ROBERT KANN.

The Illinois Oil Fields. H. A. WHEELER. Eng. Mining J., 92, 355-6. R. K.

A Report of Maikop. Anon. Gorno-Zaodskoie Delo; thru J. pétrole., 11, 98-9.

—The oil in this field is found in 3 horizons; (1) produces heavy oil in small quantities, (2) very rich in light oil, (3) light oil produced by deep drilling. The importance of the field has been exaggerated.

F. M. ROGERS.

The Katalla Oil Fields. Anon. Mining Sci., 64, 61-2.—These important fields are situated in Alaska near Controller Bay. The crude is valuable because it yields the phenominal amt. of 35-40% of gasoline. The oil belt extends along the coast for more than 100 miles.

R. E. H.

Organic Residues from Soluble Bitumen Determinations—Sulfur in Tar Residues. P. Hubbard and C. S. Reeve. Eng. Contracting, 36, 126-7.—Residues obtained from refined coal tar according to the flask method with benzene and CS<sub>2</sub>, both cold and b., showed presence of C 90.17-94.26, H 2.95-3.31, O 1.81-5.91, S 0.50-1.78%, N no trace on qual. test. Work was concentrated on S in tar residues. From 0.2 to 0.3 g. was placed in a weighed Pt cone and burnt in an 8 l. bottle according to Graefe's method, the asbestos remaining on the cone being dried, weighed and subtracted from the original sample. The results showed that with CS<sub>2</sub> the % of S increases with length of digestion and with increase in temp. of solvent. Compared with benzene extractions a short digestion with CS<sub>2</sub> produces no reaction with formation of S compds. Increasing cold digestion with benzene showed a slight increase in S and hot digestion showed no increase, reversing the CS<sub>2</sub> results.

A. J. Phillips.

The Nature of Asphalt. Anon. Mining Sci., 64, 51. R. E. H.

Good Roads in the Making. D. Basil W. Alexander. California Derrick, 4, 16.—To get rid of the large amts. of H<sub>2</sub>O contained in the crude oils used in road-making, tanks are equipped with steam heating coils placed 2-3 ft. from the bottom, and the oil is raised in temp. to a point where the H<sub>2</sub>O will drop out. A better method is to pass the oil in a fine spray into the bottom of a tall and narrow tank provided with a conical bottom, filled more than half full with water which is kept at 165-190° F. by a steam coil under the spray. The content of H<sub>2</sub>O is reduced below 1% at a cost of about 5c per bbl. This oil is then charged into a still and heated under slight pressure. The only opening in the still is a 2 in. line. The pressure holds in check the tendency to foam and "puke." The steam escapes from the small line until nearly all the H<sub>2</sub>O is removed—a point indicated by a rattling in the still. The vapor line is then opened and the distillation proceeds without trouble. When the temp. reaches 250° F., steam is turned in thru perforated pipes to help carry out the heavy oil vapors, and the distillation is thus carried on until the residuum in the still is of the required consistency. It was a former practice to reduce the residuum to a hard penetration

and flux it back, but this caused a "cracking" of the oil with formation of carbenes and other deterioration; now it is used just as it comes from the still. R. E. H.

Analyses of Mixtures (RAKUZIN). 7.

Detecting Mineral Oils (OUTERBRIDGE). 27.

Distillation Residues (MARCUSSON). 27.

Examination of Oils (DUPERTHUIS). 27.

Tests for Road Materials (PAGE). 20.

Brit., 23,125, Oct. 6, 1910. J. TANNE, Rozniatow, Galizia, Austria. Separating solid hydrocarbons, such as paraffin, ceresin, and ozokerite, from mineral oil and tar residues, by dissolving the residues in at least 0.5 and at the most in an equal wt. of CCl, the soln. being then cooled in refrigerators, the pulpy paraffin being finally sepd. out and pressed.

## 23. CELLULOSE AND PAPER.

#### A. D. LITTLE.

Brit., 29,246, Dec. 16, 1910. VEREINIGTE GLANZSTOFF-FABRIKEN, Elberfeld, Ger. Mfg. materials similar to celluloid from formyl cellulose or cellulose phosphoformate, by pptg. out said materials as a slimy mass from their soln. in HCOOH by amyl acetate, and after further giving up of the occluded solvent or pptg. agent, working them with camphor or camphor-substitutes to form elastic substances in the manner usual in the manuf. of celluloid.

Brit., 3,139, Feb. 7, 1911. A. Wohl, Haupstrasse 113, Danzig, Langfuhr, Ger. Prep. of solutions of acetyl cellulose with the use of methyl ester of HCOOH as solvent or as component of dissolving liquids.

Brit., 18,189, July 30, 1910. A. EICHENGRUN, 87 Konstanzerstrasse, Berlin, Prussia. Mfg. solid celluloid-like masses capable of being molded and pressed, by working together to a uniform mass in the presence of a solvent, but without the employment of a precipitant, acetyl cellulose, camphor or camphor substitutes, and relatively large amts. of finely powdered substances such as metal powder, minerals, (mica, asbestos, graphite) powdered glass, inorganic coloring matters and the like, the amt. of the powdered substances added being over 25%, preferably more than 100% of the acetyl cellulose employed.

# 24. EXPLOSIVES.

### C. E. MUNROE.

Explosives Laws of the Union of South Africa. Board of Trade J., June 22, 1911; Soc. Chem. Ind., 30, 835.—In effect the provisions of the Act are that no person shall import into or export from the Union any explosive unless he has obtained a permit from an inspector. No person shall manuf. any unauthorized explosives except for purposes of expt. and in quantities not exceeding 1 lb. at any one time, or 5 lb. in all. The term "explosive" is defined to mean: (a) gunpowder, nitroglycerin, dynamite, guncotton, blasting-powders, fulminate of mercury or of other metals, colored fibers, and every other substance, used to produce a practical effect by explosion or a pyrotechnic effect; (b) any fuse, rocket, detonator, cartridge, and every adaption or prep.

of an explosive; (c) any other substance which the Governor-General may declare to be an explosive.

Charles E. Munroe.

Studying the Anarchists' Bomb Scientifically. Anon. Sci. Am., 105, 100.—Describes with illustrations the lab. of the police department of Paris used for investigating and de troying bombs.

CHARLES E. MUNROE.

Fifteenth Annual Report of the Chief Inspector of Explosives for Western Australia for 1910. E. A. Mann. J. Soc. Chem. Ind., 30, 925.—The number of samples tested was 3440 (increase of 1438 over 1909) nclusive of 1409 tests for gelignite and 1757 tests for fuse. Since difficulties arose in complying with the burning speed regulations, especially with one particular brand of fuse, very stringent steps were taken to enforce compliance with the regulations and considerable quantities of fuse were condemned. In view of the exceptionally heavy importation of explosives, the quantity which it was found necessary to condemn or destroy, on any grounds, was unprecedentedly small. The importation figures for 1909 and 1910 are compared. The increase in the value of the importations over 1909 was £53,559.

C. E. Munroe.

Origin of Smokeless Powder. WALTER F. REID. J. Soc. Chem. Ind., 30, 851.—After describing his own work in gelatinizing nitrocellulose in 1881, R. states that Hartig had in 1847 dissolved nitrocellulose and used the product as a propellent.

C. E. MUNROE.

"The Explosives Report." Arms and Explosives, 19, 87.—A review of the 35th Annual Report of Home Office (see C. A., 5, 2948).

C. G. Storm

St. Vincent Regulations for Import, Sale, Manufacture, etc., of Explosives and Petroleum, 1910. Z. ges. Schiess-Sprengstoffw., 6, 159.—Government permission is required for import, sale, manuf. or possession of nitroglycerin, dynamite, guncotton, fulm nates of Hg or other metals, except in the form of cartridges for small arms, caps, fuses, etc.

C. G. Storm.

Grounding of Machinery for Powder Manufacture. A. Voigt. Giessen. Z. ges. Schiess-Sprengstoffw., 6, 204-5.—An argument to show the possibility of explosions, especially in black powder manuf., from the accumulation of static charges generated by friction of the powder material. Such charges could be readily made harmless by grounding of the machines.

C. G. Storm.

New Blasting Regulations for the Engineer Service, 1911. Von Andersch. Z. ges. Schiess-Sprengstoffw., 6, 121-3, 147-51.—A review of the new regulations for the use of explosives in the engineer service of the German Army. The following sections are discussed at considerable length: (1) Application of blasting, (2) explosives, (3) ignitions, (4) blasting with smokeless powder and blasting explosives, (5) mines, (6) precautionary measures, (7) destruction of explosives, (8) blasting equipment and tools.

C. G. Storm.

Avoiding Danger in Firing "Springing Shots." A. VOIGT. Giessen. Z. ges. Schiess-Sprengstoffw., 6, 236.—In firing successive "springing shots" to enlarge a bore hole sufficiently to receive the desired blasting charge, many accidents occur from premature discharges due to the heated walls of the hole or to smoldering fragments of fuse, etc. 30 min. is regarded as a safe interval between shots, or else water is used to cool the hole. The author suggests the use of compressed CO<sub>2</sub> for extinguishing sparks and cooling the cavity. This is easily transported in steel flasks and would ensure safety as well as great saving of time.

C. G. Storm.

Cellulose Nitrites (MARQUEYROL, FLORENTIN). 10.

Detection of Inflammable Gas (PHILIP, STEEL). 1.



CHALON, P. F.: Modern Explosives. Third edition. Paris: C. Beranger. \$5.00.

Brit., 7,625, Mar. 29, 1910. O. SILBERRAD, Buckhurst Hill, Essex Co. A fuse consisting essentially of a detonating fulminate cap sepd. only by a layer of cartridge paper from the powdered picric acid, trinitrotoluene, or trinitrobenzene contd. in the magazine of the fuse.

Brit., 2,820, Feb. 3, 1911. E. MACAULAY, Box 187, Bulawayo, Rhodesia. Composition for neutralizing or annulling the noxious gases from combustion of nitroglycerin explosives, comprizing suitable proportions of alk. salt and of an oxide of an alk. earth with or without a quantity of KMnO<sub>4</sub> or other suitable source of O.

Brit., 4,099, Feb. 17, 1911. E. I. DU PONT DE NEMOURS POWDER Co., Wilmington, Del. An explosive contg. an admixt. of butylene glycol nitrate and nitrates or nitro compds.

Brit., 9,742, Apr. 21, 1911. N. CEIPEK, 16 Kolschitzkygasse, Vienna IV, Austria. A safety explosive consisting of a mixt. of NH<sub>4</sub>NO<sub>2</sub> 88%, turmeric charcoal 6.25%, and sandal wood charcoal 5.75%.

Brit., 9,743, Apr. 21, 1911. N. CRIPEK, 16 Kolschitzkygasse, Vienna IV, Austria A safety explosive consisting of a mixt. of NH<sub>4</sub>NO<sub>2</sub> 88%, carbonized powder of curcuma root 1.05%, carbonized powder of sandal wood 0.95%, and nitrated powder of curcuma root 10%.

# 25. DYES AND TEXTIL CHEMISTRY.

#### L. A. OLNEY.

Resistance of Ingrain Colors against Resolution. W. P. DREAPER AND A. WILSON. J. Soc. Dyers Colorists, 27, 170-1.—It has already been shown by Dreaper (Ibid., 23, 667) that the relative resistance of primuline dyestuffs produced ingrain is systematically higher against subsequent resolution, than the same dyestuff applied to the fiber in ordinary manner. The authors thru continued expts. at varying temps. have shown that whenever a shade fast to soaping is desired in practice care should be taken to dye at a temp. of 95-100° for not less than 30 min. and to use a developer of a basic nature whenever the shade will allow it.

L. A. OLNEY.

Marine Fiber. A. G. Green and G. H. Frank. J. Soc. Dyers Colorists, 27, 169.—A new fiber was obtained by dredging in Spencer Gulf, So. Australia. It was probably formed from a land plant which has been submerged for some time and had undergone a natural retting process. It can be marketed in London for a little over 1 cent a lb. and practical expts. are being conducted to det. its usefulness. L. A. O.

Nature of the Combination between Fiber and Dye. J. ALEXANDER. J. Soc. Chem. Ind., 30, 517-8.—Previously pointed out by the author that after the addition of protective colloids (gelatin, etc.), to solns. of benzopurpurin, dil. acids produce color changes similar to those observed in dyed animal fibers. A dil. soln. of benzopurpurin upon addition of dil. mineral acid quickly changes from bright red to dark blue, and stronger acids coagulate the dye which settles out of soln. If gelatin is added to the soln. of benzopurpurin, dil. mineral acids give a claret-red soln., and stronger acids change the shade to chocolate brown, without causing any ppt. After examining these color changes at ordinary temps. thru the ultramicroscope the author reports that dil. benzopurpurin soln. shows a field full of ultra-microns, which from their brilliancy and motion, appear to be about 50-60  $\mu\mu$  in size. When a little acid is added the ultra-microns gather together in groups, whose motion decreases as their

size increases, until the whole of the dye is deposited in coagulated masses of bright ultra-microns. Stronger acid causes instant coagulation in large masses. When acid is added to the gelatin dye soln. no change takes place unless the acid is strong enough to cause a more or less extended agglutination of the ultra-microns into small groups of 2 or 3 which still have sufficient motion to keep afloat. The cause of the variation in the color changes produced by immersion of different fibers dyed with benzopurpurin is due, according to the author, to their difference in protective action upon the absorbed dye. After referring to Dreaper and Wilson's results with acid anthracine red the author says that a dil. soln. of that dyestuff exhibits a field, which altho showing many ultra-microns, indicates that a part of the dye is in a very fine state of subdivision, if not in true soln. Dil. acid causes no flocculation but seems to bring the dye into more perfect soln. When some of the acid is dissolved in 0.05 NNa, CO, the field shows a number of large coagulated masses of ultra-microns, which evidently represents only a part of the dye, as the soln. yields only a very slight sediment on standing. Conc. alkali causes most of the dye to flocculate and settle out of soln. This dye goes into soln. when it is heated but settles out again upon cooling.

L. A. OLNEY.

Absorption of Methylene Blue and Crystal Ponceau (Pelet-Jolivet, Siegrist).

Azomethins from Phenylisoxazolone (MEYER). 10.

Degradation of Indanthrene (SCHOLL, EDLBACHER). 10.

Dialyzability of Dyestuffs (BILTZ, PFENNING). 2.

Dichloroanthraquinones (ULLMANN, BILLIG). 10.

Vat Dyes from α-Naphthoquinone (PUMMERER, BRASS). 10.

KNECHT, RAWSON AND LOBWENTHAL: A Manual of Dyeing. Second edition. 531 pp., \$12.00.

Brit., 12,264, June 5, 1908. BAD. ANILIN- & SODA-FABR., Ludwigshafen a/R, Ger. Mfg. coloring matter of the anthracene series by condensing  $\beta$ - $\beta$ -dihalogenanthraquinone with a 1-aminoanthraquinone deriv. which is substituted, in the ring not contg. the amino group, by either the NHR or the NR, group.

Brit., 11,440, May 9, 1910. FARBWERKE VORM. M. L. & B. Míg. aminoarylacidylaminoanthraquinones dyeing from the vat and suitable for use as parent materials for the manuf. of other vat dyes, by treating with a reducing agent the condensation products obtainable by condensing an aminoanthraquinone with a nitroarylic acid chloride.

Brit., 14,448, June 15, 1910. H. LEVINSTEIN, Chemical Works, Blackley, near Manchester. Mfg. an azo dyestuff by combining diazo, tetraazo, diazo or diazodisazo bodies with glycine esters of aminonaphthol sulfonic acids, which contain the NH, and OH groups in different nuclei.

Brit., 14,559, June 16, 1910. J. HUBNER, Ash Villa, Cheadle Hulme, Chester Co. In the production of threads from cellulose, the soln. is supplied from a vessel open to the atm. at a constant head and the threads are drawn out in a funnel also open to the atm. by a single liquid.

Brit., 15,069, June 23, 1910. H. LEVINSTEIN, Chemical Works, Blackley, Manchester. Disazo or trisazo dyestuffs are obtained by combining diazotized aminoazo or secondary aminodisazo compds. of the benzene or naphthalene series with 2-halogen-5-naphthol-7-sulfonic acid.

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Brit., 15,268, June 25, 1910. BAD. ANILIN- & SODA-FABR., Ludwigshafen a/R, Ger. Mfg. coloring matters of the anthracene series by converting an aminoanthraquinone which contains 1 or more than 1 methyl group attached to a C at., into an indanthrene coloring matter.

Brit., 15,646, June 29, 1910. CASSELLA & Co., Frankfurt a/M, Ger. Mfg. azo dyestuffs by combining aromatic diazo or tetraazo compds. with m-aminophenyl-1,2-naphthimidazole-3-hydroxy-6-sulfonic acid. Disazo and polyazo dyestuffs are obtained by diazotizing in substance or on the fiber the above named dyestuffs, and combining with suitable azo compds.

Brit., 16,389, July 9, 1910. THE CALICO PRINTERS ASSN., LTD., 56 Mosley St., Manchester. Production of resist effects by printing on textil goods or fabrics a resist paste consisting essentially of 1 of the products defined as *leukotropes*, suitably thickened, and subsequently applying to the said goods a color mixt. consisting essentially of a suitable vat color with the admixt. of suitable assistants.

Brit., 19,487, Aug. 19, 1910. FARBEN. VORM. F. BAYER & Co., Elberfeld, Ger. Mfg. trisazo dyestuffs by combining the tetraazo compd. obtained drom p-aminobenzeneazo-2-amino-8-naphthol-6-sulfonic acid or from p-aminobenzeneazo-2-amino-5-naphthol-7-sulfonic acid with 1 mol. of 1,8-naphthylenediamine-4-sulfonic acid and with 1 mol. of m-phenylenediamine, m-toluylenediamine, phenol, resorcinol, cresols, m-aminophenol, m-aminocresol, or derivs. of these compds., the order of combination being immaterial.

Brit., 20,334, Aug. 31, 1910. Idem. Mfg. monoazo dyestuffs. See C A., 5, 2732.

Brit., 22,876, Oct. 3, 1910. L. CASSELLA & Co., Frankfurt a/M, Ger. In dry dyeing from a bath of chlorinated hydrocarbon, adding an aliphatic acid to an alc. soln. of the color.

Brit., 23,074, Oct. 5, 1910. B. Ronjar, 11 rue Gustave Zade, Paris. A secreting process for hair intended for hat making. See C. A., 5, 2579.

Brit., 24,820, Oct. 25, 1910. FARBWERKE VORM. M. L. & B., Höchst a/M, Ger. Producing indigo in finely subdivided forms by oxidizing by means of air or another suitable oxidizing agent the alkali salts of indoxyl or of indoxylic acid or of their homologs or derivs. substituted in the benzene nucleus, obtained as described in 22,288, of 1910, and 24,690, of 1910, under such physical conditions, or in presence of such added substances as are not technically available when the ordinary indigo melts are oxidized, owing to the necessity for recovering the alkali or because of the excess of alkali present.

Brit., 24,886, Oct. 26, 1910. *Idem*. Mfg. reddish blue to violet-blue vat dyestuffs by treating with a halogen the product obtained by condensing isatin and 7-methyl-indoxyl.

Brit., 26,739, Nov. 17, 1910. G. Voit, Furth, Blumenstrasse 13, Ger. In the production of polychrome ornamentations on transparent plastic material, inpressing a design on the outer surface of said material at the same time furnishing the recesses with a non-transparent covering, and afterwards coloring the rear surfaces with 1 or more colors.

Brit., 27,637, Nov. 28, 1910. FARBWERKE VORM. M. L. & B., Höchst a/M, Ger. Mfg. fast vat dyestuffs dyeing alizarin-red tints, by treating with a dehydrating agent a 1-α-anthraquinonyl-5-halogenarylido-2-carboxylic acid.

Brit., 1,436, Jan. 19, 1911. Soc. Anon. Pour La Fabrication Sole de Chardon-NET, Besanson, France. Bringing the double xanthate of cellulose and a base, especially soda, known under the name of viscose, into a mol. condition suitable for spinning, by incorporating with it a coagulating substance in a small proportion sufficient to bring it immediately to the desired mol. condition but not to cause any coagulation.

Brit., 8,305, Apr. 3, 1911. FARBWERKE VORM. L. DURAND, H. & Co., Basel, Switzerland. Mfg. leuco dyestuffs of the gallocyanin series. See C. A., 5, 2186.

Brit., 9,003, Apr. 11, 1911. AKT.-GES. FÜR ANILIN FABRIKATION, Berlin, Ger. Mfg. a monoazo dyestuff by combining the diazo compd. of 4-chloro-2-aminophenol-6-sulfonic acid with 2: 3-naphtholcarboxylic acid.

## 26. PIGMENTS, RESINS, VARNISHES AND INDIA RUBBER.

### A. H. SABIN, THEODORE WHITTELSEY.

Valuation of Turpentine as Siccative. P. Klason. Chem. Trade J., 48, 588; Chem. Ztg., 35, 537-9.—Distil in CO<sub>2</sub>, expose 10 cc. to air and diffused light 24 hrs.; to 5 cc. add 5 cc. of 5% soln. of cymene hydrosulfide in EtOH in 30 cc. stoppered flask, shake well, stand in dark 24 hrs., put in large flask with 100 cc. EtOH, titrate excess of cymene hydrosulfide with 0.1 N I. Test the cym. daily with I. No details are given as to surface exposed, temp., humidity, etc.

A. H. Sabin.

Chemistry of Lithography. C. HARRAP. Chem. Trade J., 48, 590.—Valuable descriptive lecture.

A. H. Sabin.

Materials of Paint Manufacture. G. B. HECKEL. J. Frank. Inst., 171, 599.

—Popular general lecture.

A. H. Sabin.

Preparation of So-called Thenard's Blue. L. Vanino. Chem. Ztg., 35, 497.—Agreeing with H. Wagner, the author corroborates McEachern (C. A., 5, 1338) on the use of various salts to correct the color in preparing Thenard's blue. V. found also that Cu, Fe and Mn interfered and that the use of Co phosphate may be avoided by using NH<sub>4</sub> alum. Various formulas are given for preparing Thenard's blue and cobalt green. Confirms McEachern with regard to cobalt green. E. J. Sheppard.

Investigations of Raw Rubber. G. FENDLER. Berlin. Gummi-Ztg., 25, 311, 351.—A reply to O. Korneck's adverse criticism (C. A., 5, 1339) of the author's modification of the nitrosite method (Gummi-Ztg., 18, 849). The author maintains his former statements regarding the impairing influence of resins in the analysis of raw rubber by the nitrosite method. The author contradicts Korneck's statement that the volumetric tetrabromide method is more desirable than the gravimetric method.

L. E. Weber.

The Production of Rubber and a New Method of Tapping the Manihot glaziovii. G. SANDMANN. Pflanzer, 7, 165-74; thru Chem. Zentr., 1911, I, 1660.—The cost of production in the Asiatic Hevea and African Manihot plantations is discussed. By a new method of tapping the Manihot, in which the milk is not coagulated on the plant, a much better grade of rubber is obtained. This method should reduce the cost of production to such an extent that the Manihot plantations can continue to compete with the Hevea plantations.

M. Heidelberger.

Rubber of Mascarenhasia elastica. Anon. Bull. Imp. Inst., 8, 346-52. T. W.

Para Rubber (Hevea brasiliensis) in Southern Nigeria. Anon. Bull. Imp. Inst., 8, 341-6.

Development of Chemistry of Caoutchouc and Gutta-percha in the Last Thirty
Years. R. Marzahn. Gummi-Ztg., 25, 1438.—Review. T. W.

Analysis of Crude Rubber and Rubber Articles. Anon. Gummi-Ztg., 25, 1445.—Reprinted from Gummi Kalendar, 1911.

T. W.

Tackiness in Rubber. Anon. India Rubber J., 41, No. 20, 12.—Review.

T. W.

Estimate of Guayule in Mexico. Anon. Gummi-Ztq., 25, 960.

Review of Chemistry of Caoutchouc and Gutta-percha for 1910. E. Mosinger. Gummi-Ztg., 25, 917, 957, 994. T. W.

Technical Reports of the Second International Rubber Exhibition in London. E. STERN. Gummi-Ztg., 25, 1570, 1604.—(1) Methods of testing rubber, (2) synthetic rubber, (3) coagulation, (4) guayule, jelutong, murac and mineral rubber.

T. W.

Regeneration of Rubber. P. Alexander. Gummi-Ztg., 25, 1280.—An address.

Guayule Rubber. H. VAN DER LINDE. J. Soc. Chem. Ind., 29, 1283-4.—The author describes the Guayule plant and its ability to withstand drought which is prevalent in the region in which it grows. The occurrence and properties of the rubber and the method of procuring it are given. The short history of the guayule industry is reviewed and a future predicted for it.

C. R. Boggs.

Determination of Caoutchouc as Nitrosite. G. FENDLER. Gummi-Ztg., 24, 1000.—Fendler criticizes statements made by Harries (C. A., 4, 1909). The high results of the nitrosite method cannot be explained as due to the nitrosites of the rubber resins, as Fendler used acetone-extracted rubber. Previous statements and figures are quoted in substantiation.

C. R. Boggs.

Chemistry of Resins (GRASSER). 29.

Detecting Mineral Oils (OUTERBRIDGE). 27.

Dimethylpentane from Dimethylcaoutchouc (RICHARD). 10.

KRÖBER, A. F.: Farbentafel streichfertiger Oel- und Lackfarben. 3 Aufl. Berlin: J. Wagner. 3 M.

PEARSON, H. C.: The Rubber Country of the Amazon. New York: India Rubber Pub. Co. 250 pp., \$3.00.

Schidrowitz, P.: Rubber. London: Methuen & Co. 8°, 290 pp., \$2.75.

Toch, M.: Materials for Permanent Painting. D. Van Nostrand. 208 pp., \$2.00.

Brit., 4,189, Feb. 19, 1910. F. E. MATTHEWS, 7 Staple Inn, County of London. Mfg. isoprene (which upon polymerization yields caoutchouc) by converting isopentane into a dibromo, or a dichloro, or a chlorobromo compd., by treating the isopentane with Br, or with Cl, or first with Cl and afterwards with Br, and subsequently removing 2 mol. proportions of the halogen acid.

Brit., 4,572, Feb. 23, 1910 *Idem*. Mfg. isoprene from the amylic alcohols, derivatives of isopentane, by replacing the OH group of such compd. or mixt. of compds. by Br or Cl and subsequently treating with Br or Cl and obtaining a dihalogen deriv., and then removing 2 mol. proportions of the halogen acid.

Brit., 4,620, Feb. 24, 1910. Idem. Mfg. caoutchough by subjecting rosin, or colophony, or equiv. matter, to a high temp. and then to fractional distillation, and

subjecting the obtained fraction consisting of, or principally of, isoprene, to condensation and polymerization so as to produce caoutchouc.

Brit., 5,932, Mar. 9, 1910. WM. H. PERKIN, The University, Manchester. Mfg. isoprene by dehydrating 1 or more than 1 of the amyl alc. derivs. of isopentane, so that it loses 2 ats. of H.

Brit., 6,486, Mar. 15, 1910. Wm. M. CALLENDER, Beauregard, Guerney. Obtaining rubber ferment from rubber-bearing plants by subjecting the latex to the action of a proteolytic ferment which is active in neutral or slightly alk. soln.

Brit., 10,056, Apr. 25, 1910. R. RANKIN, Elmbank, Hamilton, N. B. In the treatment of latex obtained from rubber and analogous trees, coating or sealing the globule or particles of caoutchouc and other matter in the latex with a coagulation medium that will effectually prevent access of air thereto, by pouring the latex into the coagulating medium. Alc., or a mixt. of alc. and me hyl alc. is employed as the coagulating medium.

Brit., 14,040, June 10, 1910. ARTHUR HEINEMANN, 60 Carlton Mansions, Portsdown, London, W. In the manuf. of isoprene by the depolymerization of turpentine oil, passing the vapors of said oil over finely divided and heated Cu or Ag.

Brit., 14,041, June 10, 1910. *Idem*. Production of caoutchouc or india rubber from isoprene by treating the isoprene with O or ozone and then heating the product until it becomes viscous.

Brit., 14,293, June 13, 1910. H. PETERS, 56 Moorgate St., London. A protective coating or varnish, consisting of about 100 g. trinitrocellulose or triacetylcellulose dissolved in about 1.5 l. of methylated ether dild. with about 2.5 l. of amyl-alc., nitrobenzene, or other solvent, and with or without the addition of amyl silicate or methyl silicate and castor oil.

Brit., 17,734, July 26, 1910. FARBEN. VORM. F. BAYER & Co., Elberfeld, Ger. Mfg. a substance capable of replacing natural caoutchouc in all its technical applications, by heating isoprene with or without the addition of agents promoting the polymerization.

Brit., 17,796, July 26, 1910. WM. CLINE, 519 West Orange St., Lancaster, Penna. An anti-oxidizing compound consisting of smoke C or C in the form of unconsumed particles of combustion combined with boiled oil, japan, or other drier, and a non-evapg. solvent such as that known as "solvertine."

Brit., 23,354, Oct. 8, 1910. J. B. M. A. COLLETAS, 15 rue Littre, Paris. A water-proof varnish or coating for building made of damp materials having for its basis, ozokerite, dissolved in white-spirit in a proportion of 10-90 parts and allowing the use of ordinary common paints on the said constructions, the paints being kept by its use from contact with the plaster, cement, armored cement, armored beton, armored stone, or any other similar material.

Brit., 25,087, Oct. 28, 1910. G. REYNAUD, 5 rue Salneuve, Paris. Mfg. India rubber by the fractional action of H<sub>2</sub>SO<sub>4</sub> at progressive degrees of conc. upon turpentine oil or other similar oil in a divided state in an absorbent material such as vulcanized rubber.

Brit., 25,850, Nov. 7, 1910. FARBEN. VORM. F. BAYER & Co., Elberfeld, Ger. Mfg. substances capable of replacing natural caoutchouc in its technical applications, by heating with or without the addition of agents promoting the polymerization, or of diluents or solvents, substances (with the exception of isoprene) of the type

: C : C - C : C in which the free valences are satd, either by H and alkyl radicles, or by alkyl radicles.

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Brit., 28,155, Dec. 3, 1910. BAKELITE GESELLSCHAFT, 33-35 Lutzowstrasse, Berlin. Mfg. a plastic body by mixing, in the presence of H<sub>2</sub>O, fibrous matter, or a pulp contg. fibrous matter, with chem. agents capable of transformation by heat into an insol. and infusible condensation product of a phenol and HCHO and forming the mass into a plastic body.

Brit., 1,980, Jan. 26, 1911. T. L. LEISHMAN, 9 Alvanly Terrace, Bruntsfield Links, Edinburgh, North Britain. A substitute for boiled linseed oil, etc., in varnishes, comprizing Tung oil, boiled soy bean oil, and a fish or equiv. non-drying oil, in various proportions.

Brit., 6,642, Mar. 16, 1911. FARBEN. VORM. F. BAYER & Co., Elberfeld, Ger. Prep. of vulcanized products from caoutchouc-like substances by treating the latter according to 15,254, of 1910, 25,850, of 1910 and 6,540, of 1911, with vulcanizing agents and processes suitable for the vulcanization of natural caoutchouc.

# 27. FATS, FATTY OILS AND SOAPS.

#### E. SCHERUBEL.

Composition and Examination of Fat Distillation Residues. J. MARCUSSON. Z. angew. Chem., 24, 1297-1302.—To distinguish between residues resulting from fats and from mineral oils instead of distilling the residue and titrating the distillate for fatty acids, the author dets. the sapon. no. of the pitchy residue. Dissolve 5 g. of pitch in 25 cc. benzene and heat 1 hr. under a reflux condenser with 25 cc. N alc. KOH. After cooling add 200 cc. 96% alc. to ppt. pitchy matters, 3 cc. of 1% phenolph. soln., 3 cc. alc. soln. of alkali blue 6b. and titrate with 0.5 N HCl. The addition of the 2 indicators gives a sharp end point. Higher results are obtained than by the old method of titrating the filtrate from the alc. pptn. of an ether soln. of the pitch. Two tables are given showing results by both methods. Differences in the character of the unsapon. portion of the residue show that with the higher sapon. no. the unsapon. is pulverulent, while with low sapon. no. it is pitchy. To sep. the oily portions of the 2 classes of residues, 10 g. of each are dissolved in 15 cc. benzene and treated with 200 cc. of petroleum ether. Filter off ppt., treat filtrate 3 times with 15 cc. conc. H<sub>2</sub>SO<sub>4</sub> and wash free from acid with H<sub>2</sub>O and alc. KOH. After evapg, the petroleum ether the asphaltfree oily portions remain. The amt. of these varies from 3.3-11.8 in the case of stearin pitch, 15.4-40.0 for wool fat pitch, and 40-60 for mineral oil residues. A table gives I no. and sapon. no. of these oily portions. To distinguish between wool and stearin pitch heat 10 g. of pitch for  $\frac{1}{2}$  hr. under reflux condenser with 50 cc. 0.5 N alc. KOH and cool. An insol. pitchy cryst. ppt. is characteristic of wool pitch; stearin pitch gives none. This ppt. is a K salt of high mol. wt. fatty acids and not a cholesterol compd. The HgBr, test for detecting mineral oil pitch in fatty pitch is carried out as follows: Dissolve 5 g. of HgBr, in 250 cc. ether with warming; heat 10 g. pitch under a reflux condenser with 100 cc. ether until soln. takes place, cool, filter, and add 25 cc. HgBr. soln. When mineral oil pitch is present a ppt. appears in ½ hr. When only small amts. of mineral pitch are present the free acids in the fatty pitch interfere and must be removed by alc. KOH before proceeding with the HgBr, treatment. The ppt. from this can be examined for SO4, Br and Hg after adding HNO2, and a HgBr2 combination shown to have been formed. The above method has given good results upon to mixts. E. SCHERUBEL.

Constants of the Cashew Nut Fat. B. H. SMITH AND E. CLARK. U. S. Dept. Agr., Bur. Chem., Bull. 137, 137-8.—The customary fat constants were detd. on the (Et) Q

extract from the cashew nut and also on the mixed fatty acids, liquid and solid acids.

H. S. Bailey.

Notes on the Analysis of Margarine. C. H. CRIBB AND P. A. E. RICHARDS. Analysis, 36, 327-33.—With mixts containing only coconut oil and butter fat it is possible to calc. the % of each present from the Reichert-Wollney and Polenski figs. if proper corrections for the volatil sol. acids due to coconut oil and for the sol. of the insol. volatil acids be applied. These factors, with a discussion of their derivation, are given. When, as is almost always the case, some other fat besides these two, is present the problem is more difficult and as yet not satisfactorily solved. Tables showing volatil sol. and volatil insol. acids obtained with various mixts. of margarine, butter fat and coconut oil are appended.

Observations on some Methods of Estimating Coconut Oil and Butter in Butter and Margarine. C. Revis and E. Bolton. Analyst, 36, 333-42.—A critical study of the methods of Polenske, Shrewsbury and Knapp (C. A., 4, 3306), Paal and Amberger (C. A., 3, 926, 963) and Kirschner, giving exact details of operation and tables of figs. obtained by these methods on 19 pure butters, and numerous mixts. of butter, margarine and coconut oil. The Kirschner modification is apparently the most satisfactory.

H. S. Balley.

The Quantitative Determination of Coconut Oil in Edible Fats. W. Arnold. Z. Nahr. Genussm., 21, 587-98.—Coconut oil can be detd. quantitatively in oils which contain no butter fat from the sapon. no. by the use of the following formulas and from the Polenske no. by reference to tables. The R.-M. no. indicates the presence or absence of butter fat. (A) Mixts. whose sapon. no. is about 197, (a) mixts. of beef tallow or lard with coconut oil. (b) margarine which contains very little or no butter fat. (1) K - 197/0.62 = % coconut oil where K = sapon. no. of the mixt. (B) Mixts. whose sapon. no. is near 190, (a) margarine prepared from coconut and sesame oils. (K - 190)/0.69 = % coconut oil. (C) Mixts. whose sapon. no. is about 194, (a) margarine prepared from cottonseed, stearin, sesame and coconut oils, (b) edible fats prepared from mixts. of cottonseed, stearin, coconut and peanut oils. (K - 194)/0.65 = % coconut oil.

Table for the Determination of Coconut Oil in Sophisticated Beef Tallow or Lard, Margarine and Prepared Edible Fats.

Coconut oil, \$.	Polenske no.		nut	Pol- enske no.	RM.	nut	enske	RM.		enske	RM. no.
3	. 0.70	1.00	IO	1.30	2.40	30	3.65	4.85	65	9.00	7.40
4	. 0.85	1.30	12	1.40	2.75	35	4.25	5.35	70	10.00	7.75
5	0.90	1.40	14	1.65	3.10	40	5.05	5.90	75	11.00	7.95
6	0.95	1.65	16	1.90	3.30	45	5.90	6.25	<b>8</b> 0	11.80	8.25
7	1.05	1.90	18	2.15	3.60	50	6.60	6.75	85	12.70	8.65
8	1.15	2.10	20	2.40	3.90	55	7.30	6.95	90	13.80	8.70
9	1.20	2.25	25	3.00	4.50	60	8.00	7 - 35	95	14.40	9.00
	C. M. CLARK.										

The Analysis of Copra. Anon. Assn. of Ger. Oil Mills. Chem. Ztg., 35, 755.—
In order to fully extract the fat from copra, shred finely in a grating mill, extract 4 hrs. with petroleum ether (b. p. 60°) in a Soxhlet app., after briefly drying the capsule, regrind with sharp sand and again exhaust 2 hrs. with the ether. The wt. of the extract after drying at 105-6° gives % of oil.

H. S. BAILEY.

Reports on Fat and Oils. T. J. BRYAN. U. S. Dept. Agr., Bur. Chem., Bull. 137, 87.—Detailed results obtained by collaboration on the Eisenschiml and Copthorne

fish oil test indicate that this is a satisfactory method for its detection. Preliminary work on the Crampton-Simon's modification of the Lieberman-Storch test for the detection of palm oil leads to the conclusion that it is unreliable, but if glacial AcOH instead of the anhydride be used only mustardseed oil and some butter fats seem to interfere.

H. S. Balley.

Abnormal Cottonseed Cake. J. GOLDING. Seifensieder Ztg., 38, 854.—Cottonseed cake having a H<sub>2</sub>O content of 18% was found to have this amt. increased to 20% in one month and to 30% upon longer standing. The oil had undergone decomp. Bacteria were isolated from the cake, but the specific one which acted upon the oil could not be identified.

E. SCHERUBEL.

Comparison of Petroleum Ether with Ethyl Ether for Determining Fat in Cotton Products. G. M. MacNider. U. S. Dept. Agr., Bur. Chem., Bull. 137, 155-7.—A review of previous work followed by a brief account of the cooperative work of the Soc. of Cotton Products' Analysts.

H. S. Bailey.

Coconut Oil of High Iodine Value. W. D. RICHARDSON. J. Eng. Chem., 3, 574.—Samples of coconut oil containing considerable quantities of coconut rind oil have abnormally high I values.

H. S. Bailey.

Contribution to the Examination of Oils. H. DUPERTHUIS. Schweiz. Gesundheitsamt. Mitt. Lebens. Hyg., 2, 65-79; thru Chem. Zentr., 1911, I, 1889.—The critical soln. temp. (TCD) of an oil, i. e., the temp. at which a definit amt. of oil dissolves completely in a definit amt. of solvent (Crismer), is of some value for distinguishing between oils if a mixt. of C<sub>2</sub>H<sub>2</sub>NH<sub>2</sub> and C<sub>2</sub>H<sub>4</sub>OH (1: 4 by vol.) is employed. 10 g. each of oil and solvent mixt. are mixed in a cylinder 3 cm. in diam. and 15 cm. long, which is then closed with a 2-hole rubber stopper thru which are passed a small stirrer and a thermometer. The cylinder is immersed in water at 50° until the mixt. clears, after which it is withdrawn, and the mixt. stirred until it again clouds, when the thermometer reading gives the TCD. A table is given of values for various oils. The TCD of 6 olive oils varied from 35.7 to 41.2°; 3 peanut oils, 42.6 to 43.4°; 2 sesame oils, 30.0 and 33.4°; poppy seed oil, 12.1°; 4 nut oils (Nussöle) 29.6 to 33.1°; colza oil, 42.3°; cottonseed oil, 26.2°. The TCD of a mixt. is about the arithmetical mean of the TCDs of the components. For the detn. of the I no. König's procedure (Chem. mensch. Nahrungs. Genussm., 3, 1 Abt. [1910] (book)) is better for oils that are not dry, while the procedure given in the "Schweizerischen Lebensmittelbuche" is better for dry oils. The expansion of an oil is represented by a curve, as it is not proportional to the temp.  $d_{150}(d_t)$  may be calculated from  $d_t$  by the formula  $d^t = d_{t'} +$ a(t'-t). a was found to be 0.000665 for 1°. M. HEIDELBERGER.

Acid Content of Coconut Oil. L. ALLEN. Chem. Rev. Fett Harz-Ind., 18, 112-3.

—In the detn. of free acid for the trade it is customary to calc. the free acid to coconut oil fatty acids. Since the mol. wt. of these vary from 200-220 according to different authorities, the author suggests that a figure be adopted in order to avoid differences.

E. SCHERUBEL.

Novel Method for Detecting Mineral and Rosin Oils in other Oils. ALEXANDER E. OUTERBRIDGE. Oil, Paint and Drug Reporter; thru Chem. Trade J., 49, 103; Chem. Eng., 14, 347-8; Metal Ind., 9, 334-6.—The method is used in detecting the presence and relative % of mineral or rosin oils in linseed oil, soy-bean oil, corn oil, lard oil, etc., and depends upon the "bloom" or fluorescence exhibited by mineral or rosin oils when exposed to certain ultraviolet rays of light. This "bloom" is enormously intensified when exposed to the light of the ordinary enclosed-arc elec. lamp. By this means a drop of mineral oil may be detected in 1000 drops of linseed or other non-fluorescent oil. So-called de-bloomed oils are detected with equal certainty.

By comparing the sample to be tested with prepared samples containing 0.1-10% mineral or rosin oil, the % can be closely estd. Beyond 10%, the sample must be diluted, as the fluorescence becomes too intense. The method is simple and rapid.

R. E. H.

Soaps from Different Glycerides. H. C. Hamilton. J. Ind. Eng. Chem., 3, 582-4.—The germicidal and insecticidal value of various soaps alone and with other active agents was detd. with the following results: B. typhosus is susceptible to the action of KOH. The combination of soap with an active agent does not always increase the germicidal value of the latter. The germicidal value of soap and cresylic acid depends on the glyceride used. The germicidal value of the alkali is not increased by any combination used in the series of tests. When combined with cresylic acid its value is 1/2 that of the alkali alone.

E. SCHERUBEL.

The Mechanism of Saponification. J. Meyer. Seifensieder Ztg., 38, 794.—Analytical investigations have not detd. with certainty whether sapon. proceeds in stages or not. Kinetic studies upon the sapon. of polyesters of polyacids and polyacohols make it certain that the hydrolysis of a glycerol ester proceeds in stages in acid and alkaline soln. In support of the theoretical investigations of Wegscheider's it has been e tablished that heterogenous sapon. also proceeds in single phases and that the intermediary products can only be detected under certain conditions.

E. SCHERUBEL.

A List of Waxes. I. D. J. LEMAL. *Metal Ind.*, 9, 341.—A detailed description of the various varieties used in the arts.

ROBERT KANN.

Apparatus for Fat Extractions (VOLLRATH). 1. Analysis of Margarine (CRIBB, RICHARDS). 12.

LEATHES, J. B.: The Fats. New York: Longmans Green & Co. 138 pp., \$1.20.

Seifen- und Pärfümerie-Fabriken. Leipzig: Schulze & Co. 8°, 178 pp., 12 M. SIMMONS, W. H., AND MITCHELL, C. A.: Edible Fats and Oils; their Composition, Manufacture and Analysis. London: Scott & Greenwood. 8°, 158 pp., 7 s., 6 d.

Brit, 15,288, June 25, 1910. G. KOLLER, 157 Sebert Road, Forest Gate, London. The incorporation of chlorinated hydrocarbon solvents and particularly of tetrachloroethane, pentachloroethane, dichloroethane, trichloroethane, and perchloroethylene into soaps and comps. of which soaps form an integral part, by means of such phenols or alcs. as are sol. in H<sub>2</sub>O and also in such chloro compds.

Brit., 28,765, Dec. 10, 1910. Wm. D. BORRILL, 1 Copley Road, Doncaster. A cleaning composition consisting of H<sub>2</sub>O 40 gals., common soda 3 stones, American black potash 2 stones, soda ash 2 stones, NaOH (95%) 1 stone, peat moss 3 stones, soft soap 7 lbs., crushed linseed 7 lbs.

Brit., 3,880, Feb. 15, 1911. F. D. THORNE, Los Angeles, Cal. A detergent compound composed of comminuted maize 100, powdered soap 50, Irish moss 0.25, borax 0.25, Na-benzoate 0.25, paraffin wax 0.50, terpineol 0.3125.

Brit., 4,586, Feb. 23, 1911. C. JOEHLINGER, 13 rue du Remorqueur, Brussels, Belgium. Mfg. solid or pasty soap by making a mixt. of olive oil, KOH, and  $H_2O$ , and another mixt. of cacao butter, KOH, and  $H_2O$ , b. these mixts. in sep. vessels

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after a certain time of rest, pouring the boiled mixts. together into a common vessel, and adding powdered pumice stone and alk. salts thereto.

## 28. SUGAR, STARCH AND GUMS.

#### A. HUGH BRYAN.

A Recently Discovered Bacterial Decomposition of Sucrose. W. L. OWEN. J. Ind. Eng. Chem., 3, 481-6.—The author discusses the formation of the gum levan. The presence of this vitiates the Clerget detn. of sucrose in raw sugar when using HCl as the hydrolyst.

A. H. BRYAN.

Determination of Dry Substances of Sugar Factory Products. K. URBAN. Z. Zuckerind. Böhmen., 35, 312-40.—Koydl, finding that sand used in drying tended to invert sucrose, advocated sugar crystals (C. A., 3, 497, 1825, 2069, 2393). Freist, on the contrary (C. A., 2, 2464; 3, 1702) maintains that with sand or sugar crystals the results are the same. Repeating Koydl's expts., the author was unable to obtain concordant results by either method; sand sometimes gave higher and sometimes lower results than sugar crystals. He explains this by stating that when sugar is used a part is dissolved and thus the evapg, surface is decreased. In the case of sand, inversion does occur with pure sucrose solns. even the the sand is previously treated with HCl, and coarse sand causes more inversion than fine sand. Powdered glass shows this to a less degree and reliable results were obtained with finely powdered limestone in the proportion of 10 to 1.5 dry substance. Treating sand by b. with NaOH soln., then HCl, then washing and drying, gave a product causing less trouble. To hasten desiccation, the use of MeOH and a preliminary drying for 3 hrs. at 60-75°, with stirring from time to time before raising the temp. to 100°, gives the best results. Increasing the amt. of sand has not much influence; the best proportion is 10 of sand to 1 of dry substance. A. H. BRYAN.

Technically Prepared Calcium Saccharate and its Behavior in Raw Juice. A. T. HOGLUND. Z. Ver. Zuckerind., 61, 375-85.—Calcium saccharate, prepared in the Steffen's process, contains generally 100 parts of CaO to 100 parts of sugar, where theoretically it should only have 49. 1 pts. The excess is uncombined CaO and Ca(OH). When mixing this impure saccharate with raw juice, the greatest amt. of trisaccharate it is possible to obtain is produced when the compd. is added (a) so that the CaO present is not slaked, viz., in solid state or else mixed with a small amt. of water or sweet water, and (b) in such amts. that the total CaO content of the resulting mixt. is 2-3%of wt. of beets worked. If, on the contrary, before adding the saccharate to the juice, it is mixed with water or sweet water to slake all the CaO, then the amt, of insol. saccharate formed is not appreciably more than if milk or dry lime had been used. When a large quantity of trisaccharate is present, the satn. is rendered more difficult. This is also due to combinations of lime and CaCO, which are gelatinous and not easily broken down by CO<sub>2</sub>. Trisaccharate can be removed by over-satn., but this is not good policy. A. H. BRYAN.

The Apparatus and Method of Working the Continuous Dry Lime Defecation in the Beet Sugar Factory, Diirnkrut. KARL KRAUSE. Oesterr.-Ung. Z. Zucherind., 40, 477-80.

A. H. BRYAN.

The Use of Large Diffusers (in the Beet Sugar Factory). GOTTLIEB MUSIL. Oestern.-Ung. Z. Zuckerind., 40, 473-6. A. H. BRYAN.

The Value of the Chemist and his Laboratory to the Engineer and Sugar Maker.

GEO. B. SCHWAB. Louisiana Planter, 47, 92.—The value of cooperation between the sugar maker, engineer and chemist in a sugar house is discussed.

A. H. BRYAN.

The Importance of the Nitrogen Content of the Sugar Beet. JOSEF URBAN. Z. Zuckerind. Böhmen., 35, 443-50.—Thru breeding expts. and estns. of the N content of beets, as well as the purity coeff. of the expressed juice, the author confirms that the N content like the sugar content is an hereditary property and does not always bear a constant relation to the sugar content. Both sugar and N contents of the beet must be considered in a rational selection of beets for breeding, as beets high in sugar do not always show high purity.

A. H. BRYAN.

Cultural History of the Sugar Beet. H. Briem. Oesterr.-Ung. Z. Zuckerind., 40, 414-21.

A. H. Bryan

The Nematode Disease of the Beet. B. NEMEC. Oesterr.-Ung. Z. Zuckerind., 40, 422-4.

A. H. BRYAN.

Influence of Salts of the Alkalies on the Optical Determination of Sucrose. C. A. Browne and G. H. Hardin. U. S. Dept. Agr., Bur. Chem., Bull. 137, 167.—Wide discrepancies are often noted between sucrose by optical methods and by gravimetric methods (copper reduction). This is often laid to the vol. of Pb ppt., the effect of acid on rotation of levulose, or to the temps. of polarization. The authors show that there is a depressing effect on the rotation of sucrose caused by the sol. salts or hydroxides of the alkali. Basic Pb(OAc), added for clarification causes a pptn. of insol. Pb salts with a liberation of free alkalies. This lowering of the rotation may be stopped somewhat by neut. the alkalinity and may be compensated for by the points enumerated above. The authors believe the allowable error of  $\pm 0.50\%$  in the detn. of sucrose and raffinose is none too great.

A. H. Bryan.

The Action of Hydriodic Acid upon Starch and Dextrin. O. DE CONINCE AND RAYNAUD. Bull. soc. chim., 9, 586—7.—Both starch and dextrin may be hydrolyzed with HI, the former requiring more of the reagent than the latter. The extent of hydrolysis is very nearly proportional to the amt. of HI used. H. S. Bailey.

The Acid Hydrolysis of Starch Granules. C. B. Durvea. J Soc. Chem. Ind., 30, 789-90.—A brief review of the literature of the acid and also enzymic hydrolysis of starch pastes is given. As a result of 2 sets of expts. (agreeing very closely), treating starch granules with 1.0% HCl at 55° and withdrawing hourly samples for exam., maltose is found to be formed in the beginning of hydrolytic cleavage. "Accompanying the production of maltose, there is a general simplification of the mol. condition of the carbohydrate material composing the granules, so that at each succeeding moment of acid hydrolysis for each mol. of maltose produced, there are formed a greater number of what might be termed starchy dextrins, and the other dextrins (including sol. starch) in the aggregate progressively simpler in constitution at each general cleavage instant, thus carrying the ideas of Musculus perhaps a little further with regard to dextrin formation."

The Use of an Alcoholic Solution of Neutral Lead Acetate for the Determination of Gums in Syrup. A. C. CHAUVIN. Mon. sci., [5] I, I, 317-8.—Reagent; Soln. of cryst. neutral Pb(AcO), in 95% EtOH (satd. at o°). Method: Add slowly from a buret 25 cc. of the reagent to 5 cc. of the gum soln. contained in a small beaker, greased with vaselin, giving the latter a gyratory motion. Filter out the ppt. on a double filter, wash free from Pb(AcO), with 95% EtOH, and from the vaselin with (Et),O, dry in air at room temp. and weigh. Nearly theoretical results were obtained with aq. solns. containing 8% gum, and with similar 50% sugar solns. H. S. BAILEY.

The Kauri Gum Industry of New Zealand. Chem. Trade J., 49, 147-8.

J. J. M.



Alcohol from Sugar Beets (ANTAL). 16.
Report on Sugar and Molasses (AGEE, HILTNER). 12.

FRUELING, R.: Examination of the Raw Materials, Products and By-products of the Sugar Industry. Brunswick: 535 pp., \$3.75.

JODIDI, S. L.: The Sugar Beet and Beet Sugar. Chicago: Beet Sugar Gazette Co. 8°, 94 pp., \$1.00.

Brit., 14,657, June 17, 1910. G. B. McMULLEN, 185 Dearborn St., Chicago, Ill. As sugar producing products, the woody constituents of sugar cane mechanically shredded into relatively thin filamentary form, containing substantially its full natural sugar content in uninverted or crystallizable form dried to a condition contg. less than 30% of moisture and sepd. from the pith portions of the plant.

Brit., 4,519, Feb. 22, 1911. C. STEFFEN, Weissgarberlande 54, Vienna, Austria. Extracting sugar from sacchariferous plants by pressing and lixiviating, the press residues from the known process being submitted directly after their pressing and while still hot to a thoro lixiviation with H<sub>2</sub>O, the ext. thus obtained being repeatedly used to treat fresh quantities of beet-roots or like sacchariferous plants, after which (either alone or with the pressed juice) it is submitted to the ordinary treatment for the extraction of sugar.

### 29. LEATHER AND GLUE.

#### WALTER J. KEITH.

A New Method of Estimating Chromium. A. Graduste. Leather Trades Rev., 44, 31.—The method is especially adapted to use on one bath chrome liquors, and depends upon the oxidation by use of alk. Br-water (Br-water added in slight excess to a satd. soln. of KOH) instead of peroxide. A quantity of liquor equiv. to from 0.3-0.5 g. of Cr is diluted to 100 cc. with distilled water, ordinary KOH added until the ppt. redissolves, 25 cc. of alk. Br added and the mixture warmed for 15 mins. on the steam bath to complete oxidation, then 15 mins. longer to remove the excess of Br. (HCl facilitates; in case the used liquor is to be analyzed, it must be boiled with 5 cc. of conc. HNO<sub>3</sub> before adding the reagents.) Sufficient HCl is added for the complete liberation of the chromic acid, and the Cr estd. by the use of 0.1 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> and starch in the presence of 10% KI. The results compare very favorably with those obtained by the peroxide method, and it is claimed that the manipulation is less difficult.

J. S. ROGERS. \*

Notes on Tannin Reactions. Georg Grasser. Collegium, 1911, 46-52.—In Jean's original iodometric method for the detn. of tannin in alk. soln., the end-point is observed by external spotting on starched filter paper. The author finds it easier to detect by letting the test flow into starch soln, in a white dish. In testing the delicacy of the reaction, the limit for gallotannic acid was found to be 0.001%, 0.02 N I being added drop by drop to the acidified tannin soln. When over 0.1% tannin was present, the starch indicator could be added directly, titrating in warm soln. Of the other tannin reagents, tartar emetic was found less sensitive, and ammoniacal Zn(OAc), still gave a slight opalescence with 0.0005%; gelatin-salt reagent is the best for old liquors high in non-tans.

W. J. Krith.

Report on Tannin. J. S. ROGERS. U. S. Dept. Agr., Bur. Chem., Bull. 137, 171.—The report gives the results of a comparison of different methods for detg.

moisture in leather. The following methods were compared: (1) Drying in a water oven at 95-98°C for 15 hrs., (2) drying in a vacuum oven at 99° for 4 hrs., (3) drying in a vacuum oven at 70° for 4 hrs., and (4) by distillation with toluene. (1) and (2) compared very well with (3), (4) gave low results and (2) gave high results. A comparison of the results obtained by the water extraction of leather following the Bur. of Chem. method and the A. L. C. A. method is given. These extractions were made on samples of leather in the following conditions; "original," "water-free," "fat-free," "water-free-fat-free," and "fat-free-water-free," in order to det. what condition of sample was best suited to water extraction, and whether previous heating or extraction of fat with petroleum ether had any effect on the water solubles. The results in all cases by the A. L. C. A. method were considerably lower than those obtained by the Bur. of Chem. method. The condition of sample best adapted for the extraction of the water solubles seems to rest between the "original" and the "fat-free" sample, both of which gave higher result than were obtained by the extraction of the samples in any other condition. It was recommended that work be continued on extraction of water solubles, detn. of fats, detn. of N on "original" and "fat-free" sample and the development of tests indicative of the wearing qualities of leather. J. S. ROGERS.

Leather Yield and Leather Analysis. W. APPELIUS AND L. MANSTETTEN. Collegium, 1911, 133-48.—Following v. Schroeder, the rendement and tannage numbers are the resp. wts. of air-dried leather and tannin resp. to 100 parts hide substance; this last is derived from analysis, 1 part N = 5.62 hide. Von Schroeder also computed an approx. rendement on green or white wt., assuming that 125 parts green hide or roo white = 28-31 dry hide (by analysis). The authors hold that the yield is not thus correctly rendered since it depends not only upon the tannage but other elements of which the analysis takes no account, such as correct statement of green wt., location of hide sample, variations in beam-house work, etc. For instance, 50 lots of hides showed rendement limits of 61.5-65% by analysis, i. e., a practically uniform tannage. By actual factory practice, these hides returned 45.7-59.3% reckoned on green wt., some single hides giving differences of 23%. It is very difficult to select an average hide sample for analysis; portions loose in texture take up the most tannin and hence return highest yield by analysis, while in practice the reverse holds, for the loose hide retains the most water, affecting the green and white wts. Numerous tabulated analyses of oriented hide samples of various tannages are quoted. In agreement with Paessler, the neck gives the best average. W. J. KEITH.

The Chemistry of Resins. Georg Grasser. Collegium, 1911, 148-50—The physical constants, distillation products, behavior with solvents, alkalies, etc. of the resins used in lacquering leather are cited, including copal, colophony, dammar and elemi.

W. J. Keith.

The Testing of Tannin Extracts. A. Gansser. Collegium, 1911, 101-6, 109-13, 177-80.—The author reports analyses and applying his "animalized" cotton (gelatin-coated, treated with HCOH) to testing the wt. and strength-giving properties of various extracts, principally of Italian manuf. These figures are not absolute nor do they at all agree with those from hide powder, but they are more concordant and are regarded as giving reliable ratios for comparison. The tests showed easily sol. tans to be the least fixed; non-tans, such as molasses, glucose and gallic acid had no influence on the absorption.

W. J. Keith.

The Valuation of Logwood. GEORG GRASSER. Collegium, 1911, 461-3.—Schreiner's hide powder method, similar to that used in tannin estimation is laborious and the results vary according to the character of the hide. The author ppts. 50 cc. of an aq. ext. (5 g. in 250 cc.) with 20 cc. of Pb(AcO)<sub>2</sub> (1: 20) and centrifuges a portion

of the product for 5 min. in a hand app. The vol. of ppt. is compared with that from a parallel standard and is proportional when the diln. reaches 0.3%. W. J. K.

Estimation of Bisulfites in Extracts. "Exangle." Leather, 3, 523; Hide and Leather, 42, 34.—The writer claims that the following method gives more accurate results than can be obtained by the distillation method (e. g., treatment with oxalic or phosphoric acid, distillation, absorption of the distillate in alkali and titration). The method is as follows: (1) Weigh 5 g. of extract, dissolve in 100 cc. of hot water, add 10 cc. 10% HCl and boil. While boiling, add b. 5% BaCl<sub>2</sub> drop by drop, stirring constantly, until pptn. is complete. Filter thru a 590 S. & S. filter, wash with hot water by decantation and on the filter until the washings show no Ba. Dry and ignite not above dull redness and weigh. (2) Dissolve another 5 g. portion of the extract in warm water, add 10 cc. 10% HCl, boil and proceed as above. (1) estimates sulfates and H<sub>2</sub>SO<sub>4</sub>. (2) estimates total S as sulfates. (2)—(1) gives bisulfites as BaSO<sub>4</sub>.

J. S. ROGERS.

Some New Tanning Agents. Quinone and Bromine Compounds. "PATHFINDER." Leather, 3, 519; Hide and Leather, 42, 36.—The author discusses the use of quinone and its derivs., and of Br and hypobromites for tanning, according to Meunier and Seyewetz. Quinone will produce a leather of very fine grain, which is resistant and permanent, but of very light wt. When used as a preliminary tanning agent and followed by a vegetable tanning material good results are obtained. The advantages claimed are the following: (1) The swollen pelt ready for tanning, undergoes this preliminary tannage, which fixes the swelling and so renders the pelt in a good condition to absorb the tannin. (2) It separates the fiber and increases the surface exposed to the action of the tannin. (3) The grain is given a nice feel which it retains thruout the subsequent operation. (4) The yield in wt. is generally increased. The advantage claimed over CH<sub>2</sub>O tannage is that the quinone-pelt compd. is permanent. A disadvantage lies in its limited solubility but this is avoided by he use of the sulfonated quinone and its Na salt. Br-water may also be used as a tanning agent; HBr is formed gradually when in contact with the pelt and causes excessive swelling. To prevent this, common salt may be added, or an alkali which produces hypobromites. This preliminary tannage may be completed by the use of chrome, alum or vegetable tanning J. S. ROGERS. agents.

The Decay of Bookbinding Leather and its Prevention. LOUBIER. Bibliothek des Kunstgewerbe-museums, Berlin. Collegium, 1910, 463-8.—The author draws largely upon the report of the special committee of the London Society of Arts, 1901-5, and includes his own observations. Bindings made since 1860 show the most decay, due to the methods of modern tanning. The pyrogallol tans, especially sumac and gall-nuts, give the most durable leather, while the catechol tans, quebracho, etc., contain acids which cause decay. Oriental leathers are short-lived. Selected coaltar dyes are just as durable as vegetable colors; care must be taken to use harmless mordants and to avoid the use of strong mineral acids. Dr. Lamb found red, blue, and black the most light-proof. Artificially grained or split leathers are too weak for binding. Russia leather is over-rated and has little endurance. Bindings should be protected from hot air, gas-light vapors and sunlight.

W. J. Keith.

The Principles of Leather Stuffing. H. G. BENNETT. Leather Trades Rev., 44, 186; see C. A., 5, 2754.—"Stuffing leather consists in the permeation of the fibrous tissues with various kinds of greases." The principal reasons for stuffing leather are given: (a) To give pliability and flexibility. The grease functionating here as a lubricant. (b) To water-proof the leather. (c) To give further tannage, by aldehydes

formed by the oxidation of the oils. (d) To give weight. It is much easier to stuff a tanned leather than a pelt, because the fibers in the tanned leather are separated to some extent during the tanning process. Oils do not penetrate tanned leather as readily as does water, owing to their higher surface tension. The author gives various methods used for lowering the surface tension of oils in order to facilitate their penetration into the leather; (1) Placing the oil in contact with another liquid. The difference between the surface tension of oil and water is less than that between oil and air. The oil is applied to the wet leather. Thus the surface tension is lowered and as the water evaporates the oil easily takes its place. This method is used in the oiling of sole and dressing leather, and is the principle of "hand stuffing." (2) By rize of temp. When a liquid is heated the space between its particles is increased and consequently the force attracting these particles is decreased, and thus surface tension is decreased. In this method the leather is either warmed and the hot grease painted on, or the leather is immersed in the hot grease. (3) Rize in temp. when in contact with another liquid. This is a combination of the two preceding methods and is called "drum stuffing." (4) Influence of dissolved substances. A soln. in contact with a liquid often exhibits lower surface tension than the solvent in contact with the same liquid. Thus the surface tension between water and oil may be lowered by dissolving certain substances in the water. When such a mixture is agitated an emulsion is formed. This process is called "fatliquoring" and is often used in combination with the other three methods. J. S. ROGERS.

Intern. Assoc. of Leather Trades Chemist's Biannual Conference in Paris. Leather Trades Rev., 44, 37.—Discussion of the proposed official methods of the analysis of leather and of Prof. Proctor's new method for color measurement.

J. S. R.

The Dyeing of Furs. "FOURRURE." Leather Trades Rev., 44, 188.—The writer describes the clearing of the furs from grease, dyeing with ursol black, with ursol DD and the furrines.

J. S. R.

Felmongering. "PULLER." Leather Trades Rev., 44, 106.

Sheep Skin and its Many Uses. "Roy." Leather Trades Rev., 44, 107.

Dressing of Hides for Enamelled or Japanned Work. FOREMAN. Leather Trade Rev., 44, 30.—The writer discusses the following steps in the dressing of hides; shaving, slicking and flatting, buffing, drumming and scouring, compoing and slicking out, stuffing and setting, and finishing.

J. S. R.

The Manufacture of Glazed Kid. Manager. Leather Trades Rev., 44, 277.—The writer describes the preliminary treatment of the skins, liming, painting with depilatory, unhairing, fleshing, washing, puering, scudding and plumping. He takes up the subject of chroming and describes the treatment for both brown and black glacés.

Colloid Chemistry and Tanning. H. R. PROCTER. Leather Trades Rev., 44, 109; see C. A., 5, 2754.

J. S. ROGERS.

Diffusion of the Tannins thru Gelatin Jelly. A. W. HOPPENSTEDT. J. Am Leather Chem. Assoc., 6, 343-51.—A gelatin soln. was allowed to set in large test tubes, half of the number of which contained a small amt. of an iron salt. Solns. of 16 different tanning materials were poured on top of the gelatin and the diffusion noted by development of color. The materials presented the following order in diffusing power: sumac (Sicily), sumac (Va.), divi-divi, gambier, chestnut ext., myrobalans, oak ext., valonea, algarobilla, hemlock bark (Wis.), hemlock bark (Pa.), hemlock ext., ordinary quebracho ext., clarified quebracho ext., mangrove bark (African), mangrove bark (Venezuelan). A number of interesting observations are noted.

CHARLES R. OBERFELL.

Brit., I,III, Jan. 16, 19II. C. HASENBRING, 3 Burggarten, Hamburg, Ger. Treatment of soft vulcanized fiber for manufacture of soles and heels for boots and shoes, by immersing the soft vulcanized fiber plates or the soles and heels cut therefrom, for from 8 to 10 mins., according to their thickness, in a bath comp. of 2 parts by wt. of linseed oil, petroleum 1, and drying the material so treated in the air after draining off the superfluous liquid.

## 30. PATENTS.

Abstracts of patents are included under the foregoing divisions of the Journal. The abstracts of U. S. Patents are prepared by Earl T. Ragan, those of the British, French and German Patents by O. D. Swett and those of Canadian Patents by Russel S. Smart, of Fetherstonhaugh & Co., Ottawa (5 Elgin St.). The abstracts of French Patents are prepared, with permission, on the basis of the abstracts in *Moniteur scientifique*, those of German patents, with permission, on the basis of the abstracts in *Chemisches Zentralblatt*.

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## CHEMICAL ABSTRACTS

Vol. 5. OCTOBER 20, 1911. No. 20

#### APPARATUS.

L. C. JONES.

Convenient Form of Phosphorus Pentoxide Tube. E. Moles. Anales soc. espan. fis. quim., 9, 214-5.—This consists of a straight tube of uniform diam. with inlet and outlet at the ends thru stopcocks. These stopcocks are of nearly the same diam. as the tube and fit in the direction of its axis, so that by their removal the tube may be readily cleaned. P.O. is held in place by plugs of glass wool. H. S. PAINE.

Handy Buret Holder. H. ROBERTS AND F. A. McDERMOTT. J. Am. Chem. Soc., 33, 1402-4.—A piece of board 50  $\times$  8  $\times$  2.2 cm. is bored with holes 2.6 cm. in diam, a little in front of the middle with 5 cm, centers, the holes being connected with the front of the board by saw cut slots 1.7 cm. wide. The burets are held in position by bored conical corks and the perforated board supported by a bracket screwed to a vertical lab. stand.

Contrivance for Catching Water Condensed on Return Coolers. L. DEDE. Chem. Ztq., 35, 723.—A glass bowl with a neck in the bottom for the cooler tube to pass thru, connection being made by means of a short rubber tube. A side-neck carries the H.O away. I. H. MOORE.

A Simple Laboratory Contrivance for Producing a Continuous Stream of Ozonized Air. L. V. LIEBERMANN. Chem. Ztg., 35, 734.—A strong test tube is passed thru a cork in one arm of a Peligot U-tube, the other arm being closed with a cork. The U-tube is placed in a battery-jar filled with H<sub>2</sub>SO<sub>4</sub> of 1.4-0.5 sp. gr.; the test tube is also filled with the acid. One Pt electrode is in the test tube, the other in the jar.

I. H. MOORE.

Improved Titration Apparatus. Burkhardt. Chem. Ztg., 35, 656; see fig.—The buret, which is filled by inclining the app., and the thick-walled glass cylinder are sep. pieces. Glycerol is used in the protective tube A. This app. is manufactured by Ges. fur Laboratoriumsbedarf m. b. H. Bernhard Tolmacz & Co., 59 Luisenstrasse, Berlin, N. W. 6.

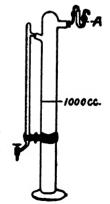
E. J. CRANE.

Gas Analysis Apparatus. F. CHARLES. Chem. Ztg., 35, 718.—A modified Bunte buret. J H. MOORE.

Stearinometer, an Apparatus for Determining the Stearin Content of Candles. F. KNORR. Oesterr. Chem. Ztg., [2] 14, 100-1; thru Chem. Zentr., 1911, I, 1569.—The app. consists of a 50 cc. buret graduated to 0.05 cc. Each 0.1 cc. corresponds to 0.2% stearin. The use of the app. depends on the following data: 1.3373 g. of pure stearin requires 50 cc. of 0.1 N KOH. The buret is connected with a reservoir containing

Zahradnik, Prag, Schulgasse.

o. 1 N KOH, and also a buret containing 1% alcoholic phenolph. Weigh out 1.3373 g. of candle, dissolve in hot alc., add 1 cc. phenolph. and titrate with 0.1 N KOH. The stearinometer will read stearin content directly in %. Can be obtained from J. SIDNEY BORN.



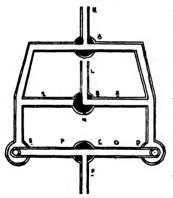
The Féry Refractometer. Chem. News, 104, 70.—A direct reading refractometer for taking  $n_D$  of oils, aq. solns., and other liquids of interest to the industrial chemist. The instrument gives, without any calc. whatever, by the simple reading of a vernier, n of any transparent liquid to an accuracy of nearly 0.0001. In spite of the high degree of accuracy the scale of the instrument extends with uniform accuracy from 1.3300 to 1.6700. Means are also provided whereby the small quantity of liquid under test (1 to 2 cc.) can be brought and maintained at any particular temp. during the measurement.

D. A. MORTON.

Thermostat for High Temperatures. M, Schuen. Elektrochem. Z., 17, 3c 1-3.—An elec. furnace for maintaining a const. temp. between 700° and 800° is described. It is of the induction type having an Fe tube as core and resistance so that the magnetic d. depends upon the temp. of the Fe, and reaches 0 at about 750° and so regulates the temp.

W. E. RUDER.

Apparatus for Continuous Fractional Distillation in a Vacuum. L. Francescont and E. Sernagiotto. Ist. chim. gen. r., Univ. Cagliari. Atti accad. Lincei, 20, II, 30-5.—The distilling app. proper consists of a 5-1. flask provided with a 2-hole stopper thru which pass the distilling head and a siphon for the introduction of the liquid to be distilled. The receiver and collector are shown in the accompanying figure. B is a 2-way, A and C 3-way stopcocks. At E and D the app. terminates in 2 concentric tubes, the larger of which are connected, by means of rubber stoppers, to the receiving flasks, while the smaller inner tubes, P and O, are joined together in the form of a T-tube and connected with the condenser at F. At M is an opening



thru which, by turning B, connection can be made between the tubes K or Z and the outside air. At L and H are connected, by their shorter arms, 2 Hg valves in the shape of inverted U-tubes with arms 50 and 90 cm. long, resp., the longer arms dipping into Hg in a suction flask joined to an aspirator. Receivers having been attached at E and D, the whole app. is evacuated by turning the stopcocks as shown in the figure; then, when the distillation is about to begin, C is turned so as to cut off P. When a sufficiently large fraction has been collected in the receiver at D, C is turned so as to shut off O and place P in communication with the condenser, and the receiver at D is completely isolated from the app. by turning A, and cautiously admitting air

thru M by turning B. It can now be disconnected and the liquid collected in it examined at once for d, rotatory power, n, etc., and a fresh receiver substituted for it. To evacuate the new receiver without interrupting the distillation, the connection with the Hg valve at H is closed with a pinchcock, the existing vacuum thus being maintained in the left-hand side of the app., then, by cautiously turning B, the fresh receiver at D is placed in communication with the pump until the pressure in it is the same as in the rest of the app., when the pinchcock at H can be removed. With this app. the authors were able to fractionate 5 1. of an essence completely in 1 week, while with the Brühl app. it required 3 months and there was a considerable loss of product. It is manufactured by Zambelli, Corso Raffaello, Turin.

CHAS. A. ROUILLER.

Agitators for Determinations of Silver, Phosphorus and Magnesium. L. W. BAHNEY. J. Ind. Eng. Chem., 3, 594-5.—Ability to remove and place bottles in ma-

chine while in motion is claimed as a distinctive feature. The clamps are of spring brass mounted on a brass disk that is operated by an eccentric having a speed of 450 R. P. M.

C. A. COLE.

An Electric Heated Sealed Tube Furnace. R. C. Benner. J. Am. Chem. Soc., 33, 1402.—The iron tubes are covered with a thin layer of asbestos paper and wound with 55 ft. of No. 22 B. S. asbestos covered nichrome resistance wire. With proper insulation from outside influence a 2 ampere, 110 volt circuit in parallel, temperatures of 150-440° may be obtained.

C. A. COLE.

Necessity of a New Saccharimeter for use in the Brewery (MOHR). 16. Accuracy of the Copper Coulometer (DEDE). 2.

#### 2. GENERAL AND PHYSICAL CHEMISTRY.

JOHN JOHNSTON.

Radiant Energy and Matter. J. J. THOMSON. Engineering, 91, 319-21, 353-4, 386-8, 421-2, 254-5, 491-2.—Reports of a series of 6 lectures at the Royal Institution.

C. H. VIOL.

Synthetic Metals from Non-metallic Elements. HERBERT N. McCov. Science, 34, 138-42.—An address.

George W. Morey.

The Teaching of Elementary Chemistry. ROBERT H. BRADBURY. J. Frank. Inst., 172, 163-72. George W. Morey.

Observation not in Conformity with the Existence of Atoms and Molecules. G. Magnanini. Modena. Gass. chim. ital., 41, I, 383-4.—The author points out the difficulty of reconciling the fact of chem. association at high temp. (as the formation of O<sub>3</sub> from O) and the idea of increased energy of the particles when the temp. is raised.

Chas. A. Roulleer.

Mendel6eff's "Cubical" Periodic System of the Elements, and the Disposition of the Radio-elements in this System. A. VAN DER BROEK. Physik. Z., 12, 490-7.—The author arranges the elements in a rectangular parallelopiped 5 spaces deep, 3 wide, 8 long, the top surface consisting of the elements from He to F along one edge, those from Ne to Cl in the middle row, and those from Ar to Mn along the front edge. The next lower layer starts with Fe, etc. This arrangement provides 120 spaces, gives place for the rare earth metals, the radioactive elements amd their decomp. products, and avoids placing Na, Cu, Ag and Au in the same vertical column. It gives 2 periods of constant size, with close agreement between theoretical and actual at. wts.

GEORGE W. MOREY.

The Principle of LeChatelier-Braun and the Reciprocity Laws of Thermodynamics. P. EHRENFEST. St. Petersburg. Z. physik. Chem., 77, 227-44.—The author gives the following as the usual statement of the LeChatelier-Braun principle: The stable equil. of a thermal system is detd. by the parameters  $a, b, c, \ldots$ , all of which are fixed except  $\rho$  and  $\sigma$ . An external influence affects  $\rho$  directly and produces a change  $\partial_1 \rho$  if  $\sigma$  is kept constant. If, however,  $\sigma$  is allowed to change also, it must change in such a direction that the abs. value of the change in  $\rho$ ,  $\partial_2 \rho$  must be smaller than when  $\sigma$  is const., i. e.,  $|\partial_2 \rho| < |\partial_3 \rho|$ . [An exam. of the references which the author gives in support of the claim that the above is the usual statement of the principle shows that the original article of LeChatelier and the text-book of Chwolson state the principle in a form similar to the above statement, but that the article of Braun and the text-books of Nernst and Weinstein give a quite different statement to which the criticisms given

below do not apply.—ABSTR.] The author then points out that this statement is not always true. For example, consider the system I vapor at const. temp. When the external pressure is increased the directly influenced parameter ( $\rho$ ) is the vol. which changes  $\delta_1 v$  if the degree of dissociation  $\alpha$  remains unchanged temporarily. The above principle requires that  $\alpha$  should change in such a direction as to make  $\left|\delta_2 v\right| < \left|\delta_1 v\right|$ , or, in other words, that dissociation should occur. As a matter of fact, association occurs which makes  $\left|\delta_2 v\right| > \left|\delta_1 v\right|$ . The author then points out that if the parameters used to det. the system are entirely intensity parameters or entirely quantity parameters, the sign of the inequality is always opposit to that given in the statement of the principle above, but in agreement with that obtained in the example quoted. If the parameters used are partly intensity and partly quantity parameters the inequality may have either sign and therefore the rule cannot be used except by careful consideration of the analogy with known cases.

The Application of the Theory of Energy Units to Physical-chemical Problems. J. Stark. Physik. Inst., Aachen. Z. Elektrochem., 17, 420-1.—Claim of priority over Nernst (C. A., 5, 2765) in the application of Planck's theory of energy units (cf. C. A., 2, 1922; 3, 398).

GRINNELL JONES.

Latent Heat of Vaporization of Liquids. Wm. C. McC. Lewis. *Phil. Mag.*, 22, 268-76.—A theoretical article devoted to the discussion of the relations of van der Waals, Dupré and Trouton. The author concludes that the latent heat of vaporization L of a liquid is connected with its compressibility  $\beta$  and the coefficient of expansion  $\alpha$  by the relation  $L = -T\alpha/\rho\beta$ . The applicability of this relation appears, however, to be restricted to normal liquids alone.

ROBT. F. EARHART.

Experimental Investigations on the Relation of the Dielectric Constants of Organic Compounds to their Composition and Structure. Dm. Dobrosserdov. Kasan. Sapiski Kasaner Univ., 1909, 1-326.—The author has detd. by the second Drude method (Ann. Physik, 8, 336), the dielec. constants for 154 generally representative organic compds. The values, which it is claimed are within  $\pm 0.5\%$  correct, together with the ds., may be found tabulated in Chem. Zentr., 1911, I, 953-6. The dielec. const. (k) is in general a distinct constitutive property. No formula for the calc. of k from that of the component elements of compds. is possible. In a homologous series k decreases continuously with increase in mol. wt.; isomers exhibit different values. The greatest differences observed for different classes of organic compds. were conditioned chiefly by the presence of a polymeric element, especially N. Changes in k brought about by the addition of the same radical to different compds. are in general different. S has a greater effect than O. The presence of many bonds, especially in ring compds., increases k.

Molecular Refraction of Organic Compounds for Rays of Infinity Great Wave Length. Dm. Dobrosserdov. Sapiski Kasaner Univ., 1909, 1-89; Chem. Zentr., 1911, I, 956.—By substituting the values of k (preceding abstr.) in the Lorentz formula, the mol. refraction for infinitly long waves has been calc. for about 280 org. compds. It was found that the value of this quantity is markedly dependent on the constitution of the compd.

E. J. Crane.

Spectrophotometric Investigation of the Opalescence of One-component Matter near the Critical Condition. W. H. Keesom. Physik. Inst., Univ. Leyden. Ann. Physik, 35, 591-8.—Measurements for light wave lengths in the region of the D line showed that the intensity of dispersed light is, for temps. up to 2.35°, above the critical temp. of ethylene, approx. inversely proportional to the difference between the temp. of observation and the critical temp.; this is in agreement with the Smoluchowski hypothesis and the author thinks it probable that the Donnan hypothesis would lead

to the same quant. relations. Ethylene at its critical d. in the region of 0.7° above the critical temp. acts optically as a disperse system whose degree of dispersion is smaller than that of a medium in which are distributed light dispersing particles which, in the sense of the Rayleigh theory, are small as compared with the light wave length. The value of the abs. intensity of dispersed light for the D line was calc. at 11.93° according to the Smoluchowski hypothesis and found to be 0.00075 as compared with 0.0007 detd. experimentally.

H. S. PAINE.

Fusion of Carbon. O. P. WATTS AND C. E. MENDENHALL. Univ. Wis. Ann. Physik, 35, 783-9; Physic. Rev., 33, 65-9.—The authors repeated the expts. of La Rosa (C. A., 5, 2012) using C rods of both American and German make and rods of Acheson graphite. The bending observed is to be ascribed to a gradual increase in plasticity rather than to melting, and the particles of drop-like structure to sublimation.

George W. Morey.

Action of the Silent Electrical Discharge on Dry and Moist Ammonia. A. BESSON. Compt. rend., 152, 1850-2.—When NH<sub>3</sub> diluted with H is acted on by the silent discharge N<sub>2</sub>H<sub>4</sub> is probably formed, as well as N and H. In the moist gas NH<sub>4</sub>OH is produced; neither of the above compds. are formed with NH<sub>3</sub>-O mixts. G. W. M.

The Constitution of Water. Jacques Duclaux. Compt. rend., 152, 1387-90. —Regarding water as a soln. of ice in hydrol, from the variation of the d. of  $H_2O$  with temp. the author cales, that the formula of ice is between  $(H_2O)_0$  and  $(H_2O)_{12}$ ; while from the density-pressure relation (calc.) it is between  $(H_2O)_0$  and  $(H_2O)_{22}$ . From a general equation representing the expansion of  $H_2O$  combined with the study of the sp. heat of  $H_2O$ , the value  $(H_2O)_{12}$  is deduced. These methods all assume that dissolved ice has the same d. as solid ice. If this is not true the mol. wt. of ice may be as low as  $(H_2O)_0$ .

George W. Morey.

Influence of Pressure and Temperature on the Electrolytic Conductivity of Solutions. FRIEDRICH KÖRBER. Göttingen. Z. physik. Chem., 77, 420-2.—Reply to Lussana (C. A., 5, 2209).

J. J.

Electropheresis of Lampblack. Albert Reychler. Bull. soc. chim. Belg., 25, 81-5; cf. C. A., 5, 641. E. J. Crane.

The Electric Energy of the Ions in Relation to the Electrolytic Dissociation Theory. F. A. KJELLIN. Stockholm. Z. physik. Chem., 77, 192-212; Archiv. Kemi. Min. Geol., 4, 1-23.—In order to account for the failure of strong electrolytes to obey the Ostwald dilution law the author attempts to show that when a soln. is diluted the work required to overcome the attraction of the oppositly charged ions is not exactly compensated by the work done by the repulsion of similarly charged ions and that the ions really obey the law  $(p + a/v^{4/3})v = \text{const.}$  instead of pv = const. Using this expression he derives for the condition of equil. between ions and unionized mols. a rather complicated expression containing 3 empirical consts., which can be selected by the aid of the method of least squares to fit the data successfully.

GRINNELL JONES.

Solutions of Sodium Aluminate. R. E. SLADE. Muspratt Lab., Univ. Liverpool. Z. Elektrochem., 17, 261-5.—Hantzsch concluded (Z. anorg. Chem., 30, 296 (1902)) from cond. measurements that Na aluminate is the salt of a monobasic acid, while Herz (Z. anorg. Chem., 25, 155 (1900)) had claimed that Al(OH)<sub>3</sub> is a tribasic acid. By addition of NaOH to a soln. containing gelatinous Al OH)<sub>2</sub>, or by the action of NaOH on Al, the author prepared solns. in which the ratio Na to Al was 1: 1. He concludes from f. p. detns. and from a discussion of the work of Russ (Z. anorg. Chem., 41, 206 (1904)) that it is the salt of a monobasic acid. The solubility product [Al(OH)<sub>2</sub>O'][H<sup>+</sup>] is given as 0.37 × 10<sup>-14</sup> at 18°.

J. JOHNSTON.

The Solubility of Aluminium Hydroxide. W. Herz. Breslau. Z. Elektro-chem., 17, 403.—A note on Slade's paper (preceding abstr.). It is pointed out that owing to insufficient stirring the Slade solns. had not reached equil. L. K. MULLER.

The Rapid Measurement of the Hydrogen Ion Concentration of Liquids. W. E. RINGER. Chem. Weskblad., 8, 293-5.—The observations of Hasselbach (C. A., 5, 1381) concerning the advantage of shaking the electrode vessel were confirmed.

E. J. CRANE.

Cosium Nitrate and the Mass Action Law. Edward W. Washburn and Duncan A. MacInnes. Lab. Phys. Chem., Univ. Illinois. Z. Elektrochem., 17, 503-9.—Biltz (Z. physik. Chem., 40, 218) calc. the dissociation of CsNO<sub>8</sub> from his f. p. data and concluded that this salt follows the Ostwald dilution law exactly. The authors have repeated the f. p. measurements, using the equilibrium method and detg. the conc. of the solns. from their elec. cond. and find  $\Delta t = (3.426 \, n - 1.862 \, n^2 + 1.373 \, n^3) \pm 0.0016^{\circ}$  where n represents the no. of moles CsNO<sub>8</sub> per 1000 g. H<sub>2</sub>O. The cryohydric point is  $-1.254^{\circ}$ . The results differ by  $0.01^{\circ}-0.02^{\circ}$  from those of Biltz. The degree of ionization calc. from these results satisfies the Ostwald dilution law between  $0.2 \, N$  and  $0.5 \, N$  when the law has no theoretical basis, but for dil. solns. where the law has a theoretical basis it fails entirely. Even when the restriction of the theory to dil. soln. is avoided by the use of the more general equation for chem. equil. (cf. Washburn, C. A., 4, 1573) in an ideal soln. in terms of mol-fractions, agreement with the theory is still lacking below  $0.2 \, N$ .

Grinnell Jones.

Studies of Chemical Affinity. IV. The Formation of Ammonium Lead Chloride. J. N. BRÖNSTED. Phys. Chem. Lab. Techn. Hochs., Kopenhagen. Z. physik. Chem., 72, 139-44; cf. C. A., 4, 1415.—The solubility curves of PbCl, and NH<sub>2</sub>Cl<sub>2</sub>PbCl, in solns. of NH<sub>4</sub>Cl of various strengths were detd. at 22°. Both of these salts can be present as solid phases in equil. with 0.52 N NH<sub>4</sub>Cl but only the double salt can exist in contact with stronger solns, and only PbCl, with weaker solns. The e. m. f. in v. of the cell PbHg NH<sub>4</sub>Cl(soln.) + PbCl<sub>2</sub>(solid) |HgCl |Hg |Hg |HgCl | (NH<sub>4</sub>Cl) solid (soln.) + NH<sub>4</sub>Cl.<sub>2</sub>PbCl<sub>2</sub>(solid) |PbHg<sub>x</sub>| was found to be 0.0302 + 0.000090(i-20) and hence the free energy (A) of the reaction NH<sub>1</sub>Cl + 2PbCl<sub>2</sub> = NH<sub>2</sub>Cl<sub>2</sub>2PbCl<sub>3</sub> (all solid) is 2780 + 8.3 (t-20) cal. By bubbling H<sub>2</sub> through a large volume of 0.05 N NH<sub>2</sub> soln. and passing the resulting H<sub>2</sub>-NH<sub>2</sub> mixture of constant comp. over platinized Pt, a reversible NH, electrode was secured which was used to measure the e. m. f. of the cell, H<sub>2</sub>NH<sub>2</sub> | (NH<sub>4</sub>Cl(soln.) + NH<sub>4</sub>Cl(solid)) | HgCl | Hg | Hg | HgCl | (PbCl<sub>4</sub>(solid) (soln.) + NH<sub>4</sub>Cl.<sub>2</sub>PbCl<sub>2</sub>(solid)) | H<sub>2</sub>NH<sub>2</sub>, in which the same electromotive reaction occurs as in the previous cell. For this cell at 18°  $\pi = 0.122$  v., hence at 18° A = 2810 cal. By a method of calc. previously described (C. A., 3, 604; 4, 2399) A at 100° is found to be 3195 cal. from the vapor press. of a satd, soln. of NH<sub>4</sub>Cl and the vapor press. of a soln. satd. with both PbCl<sub>2</sub> and NH<sub>4</sub>Cl.<sub>2</sub>PbCl<sub>2</sub>, but this result is not considered as reliable as the electromotive results. By the Helmholtz equation the heat of reaction is calc. to be 350 cal. GRINNELL JONES.

The Influence of Cane Sugar on the Accuracy of the Copper Coulometer. L. Dede. Hamburg. Z. Elektrochem., 17, 238-9.—The author recommends a soln. of 10 g. CuSO<sub>4</sub>.5H<sub>2</sub>O, 15 g. sucrose in 100 cc. of H<sub>2</sub>O as electrolyte for the Cu coulometer. This soln. prevents the deposition of Cu<sub>2</sub>O on the cathode and has a smaller solvent action on the electrodes than the usual electrolyte and thus makes it possible to use lower current d. without sacrifice of accuracy. In 14 expts. in which the c. d. varied from 0.09 to 20 milliamperes per sq. cm. and the Cu deposited from 0.1-0.4 g. the amt. of Cu deposited agreed within 0.1 mg. with that calc. from a Ag coulometer in series.

Hydrolysis of Metallic Alkyl Sulfates. W. A. DRUSHEL AND G. A. LINHART. Kent Chem. Lab., Yale Univ. Am. J. Sci., 32, 51-60.—The authors repeat the work of Kremann (C. A., 4, 2103) but do not confirm the latter's conclusions. The reaction was studied by detg. the wt. of BaSO<sub>4</sub> pptd. in successive time intervals. Ba(EtSO<sub>4</sub>)<sub>2</sub> and HEtSO<sub>4</sub> are hydrolyzed extremely slowly in H<sub>2</sub>O soln., even at 60°. The rate of hydrolysis is increased uniformly by addition of HCl, no minimum being found. Fair velocity constants were obtained on the hypothesis that Ba(EtSO<sub>4</sub>)<sub>2</sub> and HEtSO<sub>4</sub> are esters of similar stability in the presence of HCl, and the reaction for their simultaneous decomp. is molecular; better constants were obtained on introducing dissociation constants for the ester undergoing decomp. and the catalyzing acid.

GEORGE W. MOREY.

Citrophosphate Solutions. I. Homogeneous Equilibrium in Water Solution, Studied by the Cryoscopic Method. U. Pratolorgo. Milano. Atti accad. Lincei, 20, I, 812-8.—Detns. of the f. p. of solns. of citric acid (or diammonium citrate) and Ca<sub>2</sub>PO<sub>4</sub>, CaHPO<sub>4</sub>, or CaH(PO<sub>4</sub>)<sub>2</sub>, show that none of the phenomena are of such a nature that they cannot be explained as due to simple variations of electrolytic dissociation or hydrolysis of the compose, formed by double decomp. of the components.

C. A. ROUILLER.

Remarks on a Recent Investigation on the Refractive Index of Binary Mixtures. ARRIGO MAZZUCCHELLI. Atti accad. Lincei, 20, I, 752-8.—The author discusses some of the relations between different physical consts. which Schwers (C. A., 5, 614) has sought to establish and which, to the former, seem entirely too simple in form.

CHAS. A. ROUILLER.

Solubility Equilibrium between Iodine and Organic Substances. F. OLIVARI. Lab. chim. univ. Parma. Atti accad. Lincei, 20, I, 470-4.—Complete solubility diagrams of binary systems of I with various org. substances show that the solubility curves of I near its m. p. practically coincide for CHI<sub>2</sub>, Ph<sub>2</sub>N<sub>2</sub>, p-Br<sub>2</sub>C<sub>2</sub>H<sub>4</sub>, and p-C<sub>4</sub>H<sub>4</sub>-(NO<sub>2</sub>)<sub>2</sub>, but have a lesser slope for Bz<sub>2</sub>O and BzOH, probably owing to mol. polymerization (cf. C. A., 5, 1225). The curves for C<sub>4</sub>H<sub>4</sub>Br<sub>2</sub>, C<sub>4</sub>H<sub>4</sub>(NO<sub>2</sub>)<sub>3</sub>, Bz<sub>2</sub>O and BzOH show quite an extended horizontal portion, indicating the formation of 2 liquid layers. In the C<sub>4</sub>H<sub>4</sub>Br<sub>3</sub> and Ph<sub>2</sub>N<sub>2</sub> curves there is a marked inflection at conc. of 70 and 60%, resp., probably owing to the solns. passing into the neighborhood of the critical state (Eggink, C. A., 3, 139). The presence of the eutectic even in dil. solns. (5-6%) precludes isomorphism between the components beyond these limits. There is no indication of the crystn. of I complexes.

Formation of Solid Metallic Solutions by Diffusion in the Solid State. G. Brunz AND D. MENEGHINI. Univ. Padova. Atti accad. Lincei, 20, I, 671-4, 927-31.-A Ni wire 0.5 mm. in diam. was electroplated with Cu until the proportions of the 2 metals were about the same as in "constantan," then heated in a thin porcelain tube at 1000°, cooled to room temp. at intervals, and the resistance of a 20 cm. length detd. It rose from 0.0260 ohm to a const. value 0.2105 ohm after 157 hrs. heating, a value agreeing very well for that of "constantan" of the same comp. With a 0.5 mm. Au wire plated to 0.8 mm. with Cu (52.5% Au) heated to 800°, the resistance of a 14 cm. length rose from 0.0074 to 0.0336 ohm. With a 0.46 mm. Au wire plated to 0.96 mm. with Ag (62.8% Ag) at 900°, the resistance of a 20 cm. piece rose from 0.0042 to 0.0250 ohm. The larger the surface the more rapid the diffusion; the resistance of 14 cm. of a 0.075 mm. Cu wire plated to 0.625 mm. with 30 alternate layers each of Cu and Ni and containing 55,02% Cu rose in 10 hrs. at 1000° from 0,022 to 0,260 ohm. Under the microscope the wire, in which, before the heating, the different layers were quite distinct, was entirely homogeneous. Expts. at lower temp. show that the diffusion takes place with some speed at 500°. CHAS. A. ROUILLER.

Thermal Analysis of Mixtures of Cuprous Chloride with Chlorides of Monovalent Elements. C. Sandonnini. Ist. chim, gen., Univ. Padova. Att. accad. Lincei, 20, I, 457-64.—The f. p. was found to be: CuCl, 422°; AgCl, 455°; NaCl, 806°; KCl, 776°; TlCl, 429°. With NaCl the f. p. curve falls steadily to an eutectic at 314° for 75 mol. % CuCl, then rizes steadily to the f. p. of pure CuCl. With AgCl the corresponding values are 260° and 54.6%. KCl and TlCl show distinct inflections; the former at 224° for 54% CuCl, the latter at 226° for 47.5%. The eutectic and % of CuCl for that point are: KCl, 136°, 67%; TlCl, 122°, 60%. The data show that CuCl forms solid solns. in limited proportions with NaCl and AgCl, and double salts of the type CuCl.-2MCl with KCl and TlCl; also mixed crystals with TlCl (within quite narrow limits). Chas. A. Rouller.

Binary Systems of the Chlorides of some Monovalent Elements. G. Poma and G. Gabbi. Inst. chim. gen., Univ. Parma. Atti accad. Lincei, 20, I, 464-70.—Apparently entirely independently of Sandonnini (preceding abstr.) the authors detd. the f. p. curves of the systems CuCl-AgCl and CuCl-KCl with the same results, although the numerical data differ somewhat. The f. p. of the pure salt found by them is: AgCl, 451°; CuCl, 415°; KCl, 759°.

Chas. A. Rouiller.

The Binary Systems CuCl-AgCl, CuCl-NaCl, CuCl-KCl. P. DE CESARIS. Lab. chim. gen., Univ. Roma. Atti accad. Lincei, 20, I, 597-9; cf. preceding abstrs.—The results obtained agree quite closely with those of Sandonnini. Chas. A. Rouiller.

The Binary System CuBr-KBr. P. DE CESARIS. Atti accad. Lincei, 20, I, 749—51; cf. preceding abstr.—The diagram shows the existence of a compd. CuBr.2KBr. The f. ps. of CuBr and KBr were found to be 478° and 730°, resp.; at 384° CuBr undergoes a transformation. The eutectic is at 182°.

CHAS. A. ROUILLER.

Thermal Analysis of Binary Mixtures of Chlorides of Monovalent Elements. CARLO SANDONNINI. Lab. chim. gen., Univ. Padova. Atti accad. Lincei, 20, I, 758-64; cf. preceding abstrs. and Botta, C. A., 5, 2376.—The systems AgCl-NaCl, LiCl-CuCl, LiCl-AgCl were studied. The results obtained for AgCl-NaCl agree with those secured by Botta. The f. p. of LiCl was found to be 602°. AgCl-NaCl deposit mixed crystals of only 1 form and in all proportions. LiCl-AgCl form mixed crystals of 2 kinds; a gap in miscibility exists between about 16 and 50 mol. % AgCl. LiCl-CuCl give mixed crystals of 2 kinds, the gap in miscibility extending from 25 to 55% CuCl. There is a minimum in the f. p. curve for about 80% CuCl.

Crystallization Phenomena in Ternary Systems. I, II, III. Isomorphous Ternary Mixtures with a Gap in Miscibility. IV. Limiting Cases of the Gap in Miscibility N. PARRAVANO AND G. SIROVICH. Univ. Roma. Gazz. chim. ital., 41, I, 417-53, 478-89, 569-620, 621-44.—General theoretical discussion of the phenomena of solidification of isomorphous ternary systems with a gap in miscibility in the solid or liquid state.

Chas. A. Rouiller.

Influence of Neutral Salts on the Velocity of Reaction. P. POMA. Ist. Nobel, Stockholm. Gazz. chim. ital., 41, I, 353-83.—The hydrolysis at 20° of AcOEt by HCl, HBr, HI and HNO<sub>3</sub> in the presence of various neutral salts with a common anion was measured. The true H<sup>+</sup> conc. having been approx. detd. by Kay's method (Proc. Roy. Soc. Edinburgh, 22, 484), the % increase of the reaction const. due to the neutral salt was found by Arrhenius' equation (Z. physik. Chem., 4, 236). The values of this increase, for 0.0817 N AcOEt and 0.03 N HCl and salt cones. of 1, 0.6 and 0.3 N conc., resp., are given below: KCl, 50.3, 31.3, 16.0; NaCl, 47.1, 30.2, 15.5; LiCl, 45.2, 28.9, 15.7; BaCl<sub>2</sub>, 42.1, 28.1, 14.6; SrCl<sub>2</sub>, 41.9, 27.1, 14.3; MgCl<sub>2</sub>, 43.3, 27.5, 15.8. With HBr: KBr, 40.8, 25.5, 14.9; NaBr, 41.5, —, 13.1; BaBr<sub>2</sub>, 37.5, 24.0, 13.7. With HNO<sub>2</sub>: KNO<sub>2</sub>, 23.70, 15.60, 10.10: NaNO<sub>2</sub>, 24.60, 16.70, 11.00; Sr(NO<sub>2</sub>), 22.80,

17.40, 11.10. With HI at 20° the reaction was so slow and the decomp. so great that expts. were made at 30°, the results for 1, 0.75 and 0.30 N KI being 33.00, 26.00, 63.60, resp. These increases are approx. proportional to the ionic conc. of the neutral salt; apparently with those salts whose influence is not so marked the reaction const. increases more rapidly than the ionic conc., altho this is not definitly established. The increases depend to a marked degree on the nature of the anion but apparently not on that of the cation, the accelerating influence decreasing in the order: Cl, Br, NO<sub>3</sub>, I. This is not in harmony with the views of those who suppose that the influence of neutral salts is due to the formation of solvates, but can be explained by Arrhenius' theory of a chem. equil. between an active and an inactive form of the substance, an equil. which may be disturbed by variations in temp. or ionic conc.

CHAS. A. ROUILLER.

Catalytic Action of Ferric Thiocyanate. H. Colin and A. Senechal. Compt. rend., 152, 1586-7.—The oxidation of phenols by Fe(SCN)<sub>2</sub> in the presence of  $H_2O_2$  is to be attributed to 2 causes, first, the destruction of the SCN<sup>-</sup> group with the formation of  $H_2O_2$ , and second, the sp. catalytic action of Fe. George W. Morey.

Photochemical Studies. III. Oxidation of Iodoform by Oxygen. I. PLOTNIKOV. Organ. Lab., Univ. Moscow. Z. physik. Chem., 76, 743-52; cf. C. A., 5, 822-3.—In order to discover any similarity in the mechanism of the 2 reactions a number of catalysts which influence the oxidation of HI were studied in their relation to the oxidation of CHI<sub>2</sub>, using monochromatic blue light ( $\lambda = 436~\mu\mu$ ); benzene (in the presence of which there is a photochemical after-effect) was used as a solvent. Triphenylmethane and CHCl<sub>2</sub> accelerate, and crystal red retards, the after-effect; anethole and NaNO<sub>2</sub> nullify the after-effect, while anethole strongly retards the photo-reaction. The remaining catalysts (NaOAc, Cu acetate, KNO<sub>3</sub>, cyanin, cinnamic acid,  $K_2$ Cr<sub>2</sub>O<sub>7</sub>, nitrobenzene, Na butyrate, aminoazobenzene, aniline, nitrosodimethylaniline, and dimethylnaphtheurhodine) have no influence. The inner mechanism of the two reactions is therefore not identical and this difference is probably not due to the solvents used, since the character of the catalysts was the same for CHI<sub>3</sub> in benzene and in alc. soln.

Adsorption of Electrolytes by Sols. Wolfgang Ostwald. Leipzig. van Bemmelen Gedenkboek, 267-74.—By measurements of the elect. cond. of a colloidal soln. to which varying amts. of an electrolyte have been added, it is possible to demonstrate absorption on the colloid phase even when it is present as a sol. Calculation from Dumanski's data (Z. Chem. Ind. Kolloide, 1, 281 (1906)) for solns. of NH<sub>4</sub>Cl containing a constant added amt. of colloidal Fe(OH)<sub>2</sub>, shows that the relation between the quantity of absorbed NH<sub>4</sub>Cl and the conc. of the soln is of the usual exponential type, except when the conc. becomes so great as to cause coagulation of the sol. Coagulation sets in at the conc. at which the absorbed amt. becomes independent of the conc.; this is in agreement with the conclusions of Whitney and Ober (Z. physik. Chem., 39, 630 (1902)). This interpretation of Dumanski's results is in agreement with Freundlich's theory, which is based on the idea that the coagulating effect of an electrolyte is closely related to its absorption by the colloid phase. J. Johnston.

Electrolytic Colloids. W. B. HARDY. Cambridge. van Bemmelen Gedenkboek, 180-93.—The author considers "a classification based upon the view that colloidal solns. are merely special cases of ordinary mol. soln., which are peculiar solely in a wide departure from thermodynamic reversibility due to the operation of a large internal frictional constraint which springs from the limited mobility of one of the constituents." This immobil constituent may be an ion or a neutral mol.: for instance, an ion in metal and hydrosulfide sols, and in solns. of soap and certain proteins (classed therefore as electrolytic colloids) and a neutral mol. in solns. of gela-

tin or agar in water or of celloidin in ether or alc. (classed as non-electrolytic colloids). Owing to its immobility, or relative immiscibility, the constituent to the presence of which the peculiar colloidal features are due, behaves towards any internal surface which may be formed as though the latter were a membrane partially or wholly impermeable to it; this is the source of the frictional constraint mentioned above. A number of specific cases are discussed from this standpoint, but no new experimental material is presented.

J. JOHNSTON.

The Color of Silver Particles in Colloidal Silver Solutions Calculated According to the Mie Theory. ERNST MULLER. Starnberg. Ann. Physik. 35, 500-10.—The author has applied the Mie theory of turbid media, which takes account of the optical constants of the metal, to colloidal Ag solns. with the idea of explaining the multicolored radiation from colloidal Ag solns. as compared with the yellow and green tinted light from similar Au solns. Calculations based on this theory indicate that the radiation from infinitly small Ag particles is less for wave lengths over 512 μμ than from similar Au particles; for wave lengths under 512 µµ the radiation is greater from the Ag than from the Au particles and reaches an enormous max. (30 times that of the greatest radiation from Au particles) for  $\lambda = 400 \mu\mu$ , decreasing rapidly therefrom for shorter wave lengths. Data calculated for larger Ag particles, when expressed graphically with the radiation intensities for different wave lengths and the diameters of the particles as co-ordinates, show that the intensity of radiation, as well as the proportion in the violet, increases with decreasing diam. of the Ag particles, approaching a max. at  $\lambda = 400 \ \mu\mu$  for an exceedingly fine suspension. The larger particles would therefore appear red under the ultramicroscope, while particles as small as 80 µµ in diam. would appear of an intense blue color.

New Methods of Preparing Colloidal Selenium Solutions. A. POCHETTINO. accad. Lincei, 20, I, 428-33.—When Se is heated above its m. p. in a variety of solvents (e. g., retene, fluorene, phenanthrene, anthracene, naphthalene, α-naphthol, a-naphthylamine, Ph.CH., Ph.NH, Ph.N, PhOH, thymol, paraffin, etc.), the liquid assumes a red color which it retains on solidifying, although the m. and f. p. curves remain practically the same as for the pure solvent. Sometimes the solid is blue by transmitted light, and retains its characteristics after repeated fusions and solidifications, the red Se not passing into the gray cryst. form even when the mass is fused above the transition temp. of the Se. Sections under the ultramicroscope show a uniform distribution of Se particles. By dissolving these masses in liquid solvents (CS, Et,O, C,Ha, CHCla, EtOH) colloidal Se solns, are obtained showing the characteristic double color, blue by transmitted and red by reflected light; in general, they are not very stable, those in EtOH and Et<sub>2</sub>O being the most and those in CS<sub>2</sub> the least stable. The size of the particle depends on the 2 solvents used. On standing they deposit Se, usually in the red but in some cases in the black form. In an electrostatic field, the Se is deposited on 1 of 2 Pt electrodes connected with the source of potential, usually on the + pole, although with phenanthrene-C<sub>6</sub>H<sub>6</sub> it goes to the + and with phenanthrene-CS<sub>2</sub> to the — pole. Solns. in PhNH<sub>2</sub> and glycerol can be obtained directly by heating Se in them at their b. p. In the former case the soln. is orangeyellow, and on cooling to 60° a reddish suspension begins to be formed, the liquid then appearing red by reflected and green by transmitted light. Magnified 700 diameters, circular particles endowed with energetic Brownian movements are perceived. After 24 hrs. the Se is completely pptd. in the black form; in direct sunlight the pptn. takes place in a few min. If the soln. is heated to 100° as soon as the suspension appears, the liquid again clears up and becomes yellow. Very stable colloidal Se solns. can be obtained by hydrolysis of 0.2% K<sub>2</sub>SeO<sub>2</sub> solns.; if more conc. solns. are used, red Se is deposited after a few hrs. The sp. elec. cond. of 1% solns, does not change with time but with 0.3% and more dil. sons. it gradually increases.

CHAS. A. ROUILLER.

Variations of the Transparence of Quartz for Ultraviolet Light and the Dissociation of Matter. Gustave Lebon. Compt. rend., 153, 49-51.—The author explains a recent observation of Courmont and Nogier that a quartz Hg lamp became less transparent for the ultraviolet rays with use, by the theory that the inner surface of the quartz becomes covered with a thin opaque film. This film is formed with every kind of metal cathode, and can be removed by rubbing with a cloth. The author also notes that the dissociating power of light is inversely proportional to its penetrating power.

George W. Morey.

Ultraviolet Light. H. Buisson and Ch. Fabry. Marseilles. Rev. gén. sci., 22, 309–22.—A discussion of the ultraviolet spectral region, means of observation, spectroscopic app., source of ultraviolet radiations, absorption, ionization, chem. action, biological action, and solar radiation.

H. S. Paine.

Second Spectrum of Hydrogen. C. PORLEZZA AND G. NORZI. Att. accad. Lincei, 20, I, 819–28.—All the lines between  $\lambda$  6563.05 and  $\lambda$  3889.16 of the H spectrum visible with the app. used have been measured with an average possible error of 0.05 Å. u. The app. was a Brashear spectrograph with 10,000 lines to the in. and a focal distance of 3.048 m. The exposure ranged from 8 hrs. for the green-ultraviolet to 16 hrs. for the red-green part of the spectrum. In general, the results agree with those of Watson (C. A., 3, 6 and Proc. Roy. Soc. London, (A) 82, 189), though several new lines have been found and the existence of some observed only by Hasselberg has been confirmed. In all, about 692 lines have been measured. Chas. A. ROUILLER.

Magnetic Constants of Feebly Magnetic Alloys. Tullio Gnesotto and Maria Binghinotto. Univ. Padova. Nuovo Cim., 20, II, 384-441.—A detailed theoretical discussion of Meslin's method (Compt. rend., 140, 1683), which was used in these expts., and of the conditions under which it can be advantageously applied is given. The following alloys were investigated: Sn-Bi, Cd-Bi, Cb-Bi, Cd-Bi, Cd-Sn, Sn-Pb. The susceptibility of those containing Bi was in all cases less, in abs. value, than demanded by the law of mixtures. In the case of those not containing Bi, the smallness of the abs. values of the coeff. of magnetization makes the results less exact; it would appear that the coeff. is a linear function of the conc.

CHAS. A. ROUILLER.

Action of Trivalent Ions on Colloidal Systems (MINES). II. Electrolysis of Salts of Aliphatic Acids (HOPFGARTNER). 10. Fluorescence among Luminous Insects (DUBOIS). II. Photochemical Reactions (BENRATH). 10.

## 3 RADIOACTIVITY.

#### HERMAN SCHLUNDT.

A Radiation Emitted in the Interior of Incandescent Lamps. I. HOULLEVIGUE. Compt. rend., 152, 1240-3.—For the purpose of making an investigation of the bluish glow which is sometimes seen in an incandescent lamp maintained at low voltage, a side tube was connected to a 16 c. p. lamp, and the whole evacuated to  $\frac{1}{1000}$  of a mm. In the interior of the side tube was placed a diaphragm with an orifice 6 mm. in diam. Under these conditions, and at a voltage of 75-80 volts, a glow filled the whole lamp, and penetrated through the orifice into the side tube forming a pencil of light 6 to 7

cm. in length. The length of the pencil increased with the temp. of the filament, and decreased rapidly as the pressure in the app. was slightly increased. Contrary to what was expected, the pencil of light suffered no deviation under the action of a magnet. When an electrode was placed in the side tube, and a difference of potential of 150 volts maintained between this electrode and the mean potential of the filament a more brilliant glow was obtained, while at the same time a current passed from the electrode to the filament. In each case the glow gave a very complete spectrum of Hg, indicating that it is caused by the ionization of the residual Hg left in the lamp by the rays given off by the filament. Similar results could not be obtained with a W filament.

Cathode Rays Produced in the Interior of Incandescent Lamps. L. HOULLEVIGUE. Compt. rend., 152, 1846-9.—In continuing the study of the rays evolved from an incandescent C filament (see preceding abstract) a new app. was devized which consisted of an incandescent lamp joined by means of a side tube 1 cm. in diam. to a receiver of approx. the same size as the lamp. Within the connecting tube was placed a helix of fine Pt wire which could be maintained at a potential of 100-200 volts. The whole app. was then evacuated until only traces of Hg remained. When the filament was made incandescent under these conditions, a well defined beam of rays, which like the cathode rays, was very readily deflected by a magnet, penetrated through the connecting tube into the receiver. In addition to the electrons which thus formed the parallel beam of rays there also existed in the receiver scattered electrons which moved about in the manner of gas mols., and which constituted the non-deviable beam of rays described in the previous article. Different methods for calculating the velocity of the electrons in the parallel beam gave very different results. This was shown to be due to an opposing potential set up by the electrons themselves which had the effect of lessening their velocity as they passed into the receiver. W. H. R.

Radioactivity as a Kinetic Theory of a Fourth State of Matter. Anon. Engineering, 91, 226-7.—A report of a lecture given by W. H. Bragg at the Royal Institution. In the main a comparison of the movements of corpuscular radiations and mols. of gases.

C. H. V.

German and French Units of Measurement for Radioactive Emanations. A. JABOIN AND G. BEAUDOIN. J. pharm. chim., [7] 1, 497-9.—As a mean of 3 detns., the French mg.-min. was found equiv. to 7000 v. or German units. The adoption of an international unit is urged.

V. K. Chesnut.

The Mean Depth at which Röntgen Rays Originate within a Silver Target. W. P. DAVEY. J. Frank. Inst., 171, 277-85.—Ham (C. A., 4, 1425) has given a method by which he was able to det. the mean depth beneath the surface of a lead target at which the Röntgen rays originate, when the target is struck by cathode rays produced at various potentials. The author, using Ham's method, detd. for a silver target the mean depth at which for a given potential Röntgen rays originate in the target. This depth was found to be proportional to the potential employed in generating the cathode rays. At 1700 v. it is 0.000092 cm. and at 10,000 v. 0.000054 cm. for Ag.

C. H. V.

Methods of Measurement and Standards of Radioactivity Employed in Biological Investigations. S. LOEWENTHAL. Physik. Z., 12, 143-7.—An address given at the International Congress of Radiology, Brussels, Sept., 1910. The different standards in use for expressing the activity of mineral waters are surveyed critically. The author proposes a new standard for gases and mineral waters, namely, the quantity of Ra Em. produced by 1 mg. of RaBr<sub>2</sub> per sec.

H. SCHLUNDT.

A New Type of Electroscope for Radioactive Measurements. H. GREINACHER.

Physik. Z., 12, 209-14.—Instead of the usual cylindrical, rectangular, or parallel plate chambers used for expts. in radioactivity, the ionization vessel described is hemispherical in form, with the usual central rod as an electrode, which is situated at the pole of the sphere. The chamber walls are of Al, on account of the feeble secondary radiations it emits. Several different forms of electrode were tested with  $\alpha$ ,  $\beta$ , and  $\gamma$  rays of different intensities to ascertain the relation between shape of electrode and potential required to obtain sath, current. The simple rod electrode was found to answer for most purposes. The instrument was made by Günther and Tegetmeyer, Braunschweig.

The Beta-Rays from the Active Deposit of Thorium. O. v. BAEYER, O. HAHN AND L. MEITNER. Physik. Z., 12, 273-9.—The active deposit of Th contains two products, Th A and Th D, that emit  $\beta$ -rays. By resolving the  $\beta$ -radiation of the active deposit into a magnetic spectrum, the photograph shows distinct discontinuity. Knowing the strength of the magnetic field employed and the radius of curvature of the different rays their velocities can be calculated. The  $\beta$ -rays from Th D are deflected least and constitute a band representing velocities between 0.93 and 0.95 that of light.  $\beta$ -rays from Th A are more homogeneous, but consist of four distinct lines. The ray whose velocity is 0.63 that of light is the most active photographically. fainter rays have the velocities 0.72, 0.34 and 0.28 that of light. Absorption expts. show that the  $\beta$  rays whose velocities are less than 75% the velocity of light are slowed down about 10% in their passage through 0.045 mm. of Al, the emergent rays remaining quite homogeneous. The more penetrating  $\beta$ -rays, those of Th D, however, are reduced in velocity only very slightly in their passage through matter, and emerge less homogeneous than the entering beam probably on account of scattering. The expts. do not support the view previously expressed by Hahn and Meitne that the rays emitted by a radioactive transformation product are characterized by one definit velocity. H. SCHLUNDT.

The Complex Nature of Radium C. K. FAJANS. Univ. Manchester. Physik. Z., 12, 369-78.—Hahn and Meitner (C. A., 4, 1266) recently reported that Ra C consists of at least two products: Ra C, with a half period of 19.5 min, and Ra C, half period between 1 and 2.5 min. The expts. described (1) confirm the conclusion of Hahn and Meitner regarding the complex nature of Ra C; (2) establish the nature of the rays emitted by each of the components; and (3) offer experimental evidence of a radioactive series splitting and thus forming two distinct series of disintegration products. Active deposits of pure Ra C were prepared by the method of Lerch: The active deposit obtained on the walls of a glass tube containing a relatively large quantity of Ra Em. was dissolved in conc. HCl and after an hour, when the Ra A present had decayed to 10-6 its initial quantity, a Ni plate was immersed in the boiling soln. for 10 min. Ra C is deposited on the plate while Ra B remains in soln. By exposing a metallic disc near the active deposit of Ra C, recoil atoms are deposited on the disc. These exposures were made in an elec. field and at atm. pressure. The complex nature of the recoil product is evident from the decay curves of the total activity. The recoil deposit consists of Ra C,, Ra D (rayless), and Ra C1. The Ra C1 always present in the recoil deposit can be accounted for if Ra C1 is slightly volatil, or if a few Ra C atoms sputter over onto the recoil disc as the recoil atoms of Ra D pass thru the superimposed layers of Ra C on the Ni plate. The element, Ra C1, in the recoil product whose half period is 19.5 min., was found to be identical in properties with Ra C. Ra C, has a half period of 1.38 min., and emits only  $\beta$ -rays, which corrrespond fairly closely in penetrating power to the  $\beta$ -radiation from Ra C. Its radioactive constant is 0.0084 sec-1. Assuming an exponential law for the absorption of homogeneous  $\beta$ -rays, and the absorption coeffs. of Schmidt (C. A., 1, 270) for the two  $\beta$ -rays of Ra C, the absorption expts. performed with Ra C<sub>2</sub> further lead to the conclusion that the  $\beta$ -rays from Ra C<sub>2</sub> are complex. The  $\alpha$ -rays of Ra C are assigned to Ra C<sub>1</sub>. The quantity of Ra C<sub>2</sub> in the recoil deposit as found by comparison of its  $\beta$  activity with that of the Ra C in the source was approx.  $^{1}/_{20000}$ . The quantity of Ra D, on the other hand, that recoils upon the receiving plate was about 25% of the total Ra D atoms formed. The experimental results do not find a satisfactory explanation on the single chain theory of successive disintegration products. The results obtained, however, all follow by assuming that Ra C<sub>1</sub> produces directly both Ra C and Ra D, and thus giving the Ra series of disintegration products the form: Ra

and Ra D, and thus giving the Ra series of disintegration products the form: Ra 
$$\longrightarrow$$
 Ra Em.  $\longrightarrow$  Ra A  $\longrightarrow$  Ra B  $\longrightarrow$  Ra C.  $\longrightarrow$  Ra D  $\longrightarrow$  Ra E  $\longrightarrow$  Ra F.

main line of descent appears to be from Ra C<sub>1</sub> to Ra D; the branch line Ra C<sub>2</sub> may be the genesis of the actinium family. An experimental study of the quant. division of Ra C<sub>1</sub> into Ra C<sub>2</sub> and Ra D is in progress. An appended note gives the results of expts. conducted jointly by the author and W. Makower in which the recoil products of Ra C were obtained in a vacuum of 0.003 mm. The recoil deposit obtained in this way is about 50 times as active as when the exposures are made at atm. pressure, but the properties of the deposit are essentially the same.

H. SCHLUNDT.

The Recoil of Ra C in Vacuum. K. Fajans and W. Makower. Physik. Z., 12, 378; cf. preceding abstract. H. Schlundt.

Beta-Rays from Radium D. O. v. Baeyer, O. Hahn and L. Meitner. Physik. Z. 12, 378-9.—Hitherto Ra D has been regarded as a rayless product of the Ra series of elements. The ordinary electroscopic measurements have failed to detect any rays from Ra D. A very soft  $\beta$  radiation would readily escape detection by the electroscopic method. By resolving the radiation from a Ra D + Ra E + Ra F prep. into a magnetic spectrum a photograph was obtained showing discontinuity, and two of the lines obtained are new and are doubtless due to Ra D. The velocity of the  $\beta$ -rays from Ra D was detd. and found to be 0.37 and 0.31 the velocity of light.

H. SCHLUNDT.

The Effect of Electric and Magnetic Fields on the Charge Acquired by Polonium and the Penetrating Power of the Delta-Rays. Fr. HAUSER. Munich. Phys. Lab. Polytech., Inst. Physik. Z., 12, 466-76.—Since Po emits  $\alpha$ -rays, i. e., + charged particles, a well insulated specimen of Po should acquire a — charge. It has, however, been shown by J. J. Thomson and others, that Po also emits slow moving — particles,  $\delta$ -rays, and in greater quantity than the  $\alpha$ -particles expelled, so that the specimen actually assumes a + charge, and a receiving plate above the Po acquires a — charge. The author detd. (1) the potential required that will prevent the  $\delta$ -rays from leaving the plate coated with Po; (2) the penetrating power of the δ-rays; (3) the number of  $\delta$ -particles emitted for every  $\alpha$ -particle expelled; and (4) that the — charge acquired by Po in strong magnetic fields is opposit and equal to the + charge carried off by the expelled  $\alpha$ -particles. One of the special forms of app. used in the expts. is figured and described, and most of the experimental results shown by graphs as well as given in tabular form. To minimize the disturbing effects of ionization all the expts. were conducted in very high vacuums. Results: Negative potentials applied to the Po covered disc have no effect on the rate at which the Po acquires a charge thus showing that the number of  $\delta$ -rays that accompany the radioactive process is independent of the potential. Increasing the + potential decreases the number of  $\delta$ -rays that leave the plate and the number returned to the plate by the action of the electric field can be expressed by a simple exponential function of the applied potential. A potential of +24.5 v. returns all the  $\delta$ -rays to the Po plate. The penetrating power of the  $\delta$ -rays falls between  $^{1}/_{7}$  and  $^{1}/_{11}$  that of the  $\alpha$ -particles of Po. For the specimen of Po used it was found that about 60  $\delta$ -rays were emitted per  $\alpha$ -particle expelled. Each  $\alpha$ -particle also produces secondary rays from the brass electrode in which it is absorbed, and approx. 17 secondary rays are produced per  $\alpha$ -particle shot into the electrode. It was also demonstrated that the — charge conveyed away by the slow moving electrons, the  $\delta$ -rays, when no magnetic field is applied, is equal and opposit to the + charge acquired by the Po plate.

H. SCHLUNDT.

The Determination of Radium Emanation by means of Ionization Currents. W. S. Tirow. Physik. Z., 12, 476-80.—Using the gases and water of a thermal spring in the village of Nowaja Bièlokuricha, in West Siberia, as a constant source of Ra Em., the ionization current per l. of gas or water was detd.. (1) with the app. of Elster and Geitel according to the method of Mache; and (2) the app. and method of Schmidt. Working with the spring gases, the values of the ionization current for several detns. with each form of app. show a satisfactory agreement altho the mean value obtained with the Schmidt app. exceeds that of the E. and G. app. by 1.7%. When spring water was used in the detns., the av. value of the ionization current with the Schmidt app. exceeded that obtained in the E. and G. app. by 13.4%. This difference exceeds the error of expt., and is attributed to the existence of more parasitic ions in the Schmidt app., the vigorous shaking of the water and air being favorable to the formation of additional temporary ions. The results with water thus confirm the conclusion of Randall (Chem. Ztg., 34, 1910), that the ionization currents produced by the same quantity of Ra Em. when sepd. from solns, and introduced in different forms of electroscopes generally furnishes different values, in abs. units, for the ionization current, and that caution must therefore be exercised in using such values in comparing the radioactivity of mineral waters. H. SCHLUNDT.

Radioactivity as a General Property of Matter. Th. Wulf. Physik. Z., 12, 497-500.—The fact that the at. wts. of 20 of the first 25 elements of the periodic system differ by 4, i. e., their at. wts. are expressed by the formula 4n or 4n-1, where n is an integer, and that nearly one-half the remaining elements have at. wts. to which the formulas apply, is offered as a suggestion that all elements may be regarded as disintegration products of elements of high at. wts., the He atom or  $\alpha$ -particle standing as the fundamental unit of atomic structure.

H. Schlundt.

The Penetrating Radiation Observed during Balloon Voyages. A. Gockel. Freiburg, Switzerland. Physik. Z., 12, 595-7.—During two balloon voyages, one from Zurich, the other from Bern, the author made ionization measurements in a closed vessel using a Wulf electrometer. The results obtained during previous voyages (cf. C. A., 4, 2607) were confirmed, namely that the penetrating radiation present at high altitudes is greater than can be accounted for on the assumption that the penetrating radiation in the upper regions of the atm. is caused by radioactive products arizing from the earth's crust.

H. Schlundt.

Investigations on the Radium Content of Rocks. II. E. H. BÜCHNER. Amsterdam. Proc. K. Akad. Wetenschappen, 1911, 818-9 (English).—Measurements of the amt. of Ra in 11 specimens of sedimentary rocks of different geological periods (four marble, six limestone, one chalk) showed, with the exception of one carboniferous limestone containing 3 times the mean quantity of Ra, very little individual difference from the mean quantity  $(1.4 \times 1^{-12} \text{ g. Ra per g.})$ . This mean is in good agreement with the mean of the values for limestone found by other observers, but is less than that of Joly's data (3.3). Excluding granits, which have a high Ra content, and of which many have been investigated, the mean of sedimentary rocks is somewhat higher than the mean for igneous rocks.

Ionization of Heavy Gases by X-Rays. R. T. Beatty. Proc. Roy. Soc. London, (A) 85, 230-9.—When X-rays pass thru AsH<sub>2</sub>, SeH<sub>2</sub>, or Ni(CO)<sub>4</sub>, a large part of the ionization produced is due to the production of corpuscular rays. By lining the ionization chamber with a film of As or Se, it was found that the corpuscular radiation is produced in the same quantity per g. of substance whether the substance is in the solid state or in the gaseous condition combined with H. The ionization in SeH<sub>2</sub> was studied more closely and the quantitative results obtained show that more than 50% of the total ionization is due to corpuscular rays.

H. SCHLUNDT.

The Variation of Ionization with Velocity for the  $\beta$ -Particles. W. Wilson. Proc. Roy. Soc. London, (A) 85, 240–8.— $\beta$ -Rays from Ra Em. contained in a thin-walled glass vessel, were bent around thru 90° by means of a magnetic field of known strength, thus giving a fairly homogeneous beam, whose velocity could be calculated. The  $\beta$ -rays then passed into a heavy Cu ionizing chamber, where the ionization was measured by means of an electroscope. The increase in ionization due to reflection of the rays from the walls was detd. by allowing the beam to pass first into a vessel made of very thin Al leaf, the ionization being measured, and then into the same vessel covered with a heavy Cu casing. The author's measurements lead to the following conclusions: (1) the ionization produced per cm. by particles in free air varies inversely as the square of the velocity between the limits examined (v. = 1.24 to 2.90  $\times$  10<sup>10</sup> cm. per sec.), and (2) the ionization in a thick Cu vessel is not connected with the velocity by any simple power law but is approximately given by I = K(C-v) where K and C are constants and v is the velocity of the  $\beta$ -particles.

The Path of an Electron in Combined Radial Magnetic Field and Electric Fields. H. S. Allen. Proc. Roy. Soc. London, (A) 85, 257-62.—In the course of an investigation with a focus tube, in which the anti-cathode was a magnetic pole, some interesting observations were made with regard to the distribution of the fluorescence on the walls of the tube when the magnetic field varied. As the intensity of the magnetic increased a cone of blue light shown from the magnetic pole. To explain the various changes observed the spiral path of an electron in a radial magnetic field was investigated mathematically, and was found to lie on a right circular cone whose vertex coincides with the magnetic pole. This result continues to hold when an elec. field with its lines of force radiating from the same point is superimposed on the magnetic field.

H. SCHLUNDT.

A Method of Making Visible the Paths of Ionizing Particles through a Gas. C. T. R. WILSON. Proc. Roy. Soc. London, (A) 85, 285-8.—The tracks of individual  $\alpha$ - or  $\beta$ -particles, or ionizing rays of any kind, thru a moist gas were made visible by condensing water vapor upon the ions set free, a suitable expansion app. being used for the purpose. For eye observations a horizontal stratum of air in the cloud chamber was illuminated by a suitable source and a condensing lens; for photographing the clouds a Leyden jar discharge thru Hg vapor at atm. pres. was employed. In the absence of ions the clouds formed with large expansion  $(v_s/v_1 > 1.38)$  showed a uniform distribution of drops. Preliminary photographs are reproduced of the characteristic clouds formed on ions due to  $\alpha$ -rays and to X-rays. The method furnishes a new experimental weapon for attacking the problem of ionization produced by different types of radiations.

H. SCHLUNDT.

Energy Transformations of X-Ray. W. H. BRAGG AND H. L. PORTER. Proc. Roy. Soc. London, (A) 85, 349-65.—In accounting for energy expenditures of  $\alpha$ -,  $\beta$ -,  $\gamma$ -, or X-rays the authors assume that no energy yield is obtained from the internal store of energy in the atoms of the substances placed in the path of the rays. X- and  $\gamma$ -rays are assumed to be corpuscular in nature. No X- or  $\gamma$ -ray spends energy in its passage through matter; the only way in which the existence of such rays is made manifest is

thru their replacement by swiftly moving electrons which ionize the gas thru which they pass. The single X-ray disappears as such, and in its place is a cathode ray, an electron moving with energy inherited from the X-ray. Ionization by X-rays is thus an indirect process. The secondary X-rays stimulated by a primary beam are not considered an exception since the secondary rays are characteristic of the radiator and can only be brought into existence by a primary beam of like or greater energy. The expts. were undertaken to show (1) that the ionization of the secondary X-rays is caused by cathode rays that follow the disappearance of the secondary X-rays, and (2) that the former should alone be the cause of the ionization. The secondary X-rays from Sn, being relatively energetic, were used in the expts. The cathode radiation which the Sn X-rays produced in different substances was measured and the amts. compared with the absorption coeffs. of the substances for the Sn X-rays. From these results it is possible to est. the amt. of cathode radiation actually produced in a given wt. of metal thru the absorption of a given quantity of X-radiation. The difficulties met with and the approximations made in finding the est. are discussed, and, in particular, the lack of accurate information on the absorption of cathode rays of various speeds. The results finally obtained support the deduction that the production of cathode rays is caused by the disappearance of X-rays. Assuming that Sn X-rays produce equal cathode radiations in O and in Ag for equal expenditures of energy, it is then shown that when X-rays pass thru O the cathode radiations in the gas are sufficient to account for all the observed ionization, so that there is nothing left for the X-rays to do. This agrees with the second deduction, that the cathode rays alone cause the observed ionization. H. SCHLUNDT.

The Deviation of Canal Rays. M. MOULIN. Radium, 6, 78-9.—A reply to a note by J. Becquerel (Radium, 6, 45-7) who claimed to have produced in a Crookes tube a beam of positive electrons. The author thinks the condition of the expt. too complicated to allow of a simple interpretation of the effect of a magnet on the beam, and says the introduction of the hypothesis of positive electrons is unjustified.

C. H. VIOL.

Recoil Phenomena and the Final Product of Radium. J. McLennan. 6, 245-6; Nature, 80, 490-1 (1909).—In accord with Rutherford's theory of recoil, the author thinks in the case of polonium that the final product should be subject to the recoil phenomenon when the  $\alpha$ -particle is emitted. V. E. Pound, working in the author's laboratory, found evidence of this when an insulated Cu plate "A" of about 5 sq. cm. surface covered with a polonium deposit was placed in an evacuated vessel, opposit a second insulated Cu plate, "B", which was connected to an electrometer to measure the charges acquired by plate B, when elec. and magnetic fields were applied. With moderate elec. and magnetic fields the charge curves can be analyzed to show 3 kinds of radiations which enter into the charge acquired by plate B, viz., α-rays from plate A, easily absorbed, δ-rays from A, and an easily absorbed secondary radiation from plate B, consisting of negative charged particles. With higher magnetic fields and plate A highly positively charged, the plate B was found to take up an increasing positive charge. This was thought to be due to a heretofore undetected negative radiation (supposed to be the "recoil atoms") which are deflected by the very strong magnetic field. When plate A is neutral or negatively charged, the application of a magnetic field failed to give any indication of this radiation.

C. H. V.

Concentration by means of Carbon at Low Temperature of the Radium Emanation of the Gas of Boraciferous Soffioni. C. PORLEZZA AND G. NORZI. Ist. chim. gen., Univ. Pisa. Atti accad. Lincei., 20, I, 932-4.—The gas from the Larderello soffioni, freed from H<sub>2</sub>S and CO<sub>2</sub>, was passed over 5 g. C at —77°. At the 17th 1 the gas issuing

from the absorber had 50% of its original activity, when the process was suspended, the C being considered as satd. The original activity of the gas being 200,000 volthours, (and assuming that 500 cc. are absorbed by the C) that of the absorbed would be 6,800,000 volthours.

CHAS. A. ROUILLER.

Radioactive Tufa of Fiuggi. Occluded Gas. Radium and Uranium Content. C. Porlezza and G. Norzi. Ist. chim. gen., Univ. Pisa. Atti accad. Lincei, 20, I, 935-9.—The occluded gas of Fiuggi tufa (about 25 cc. per 200 g. rock) consists chiefly of CO<sub>2</sub> and O, with only minute amts. of He. The Ra content, detd. by Strutt's method, is  $5 \times 10^{-12}$  g. per g. rock. The calc. amt. of Ur would be  $0.76 \times 10^{-5}$  while the amt. experimentally obtained by Patera's method (Classen, Ausgew. Meth. anal. Chem., I, 659) was  $0.676 \times 10^{-5}$ , but the analysis is so difficult that not much stress can be laid on this last value.

Condensation of Radium Emanation. A. LABORDE. Radium, 7, 294-5.—When Ra Em. is condensed in a tube immersed in liquid air, and the temp. allowed to rize, the temp. at which it begins to be evolved is 20° lower with a glass tube than with a metal tube. Further, when a current of air carrying Em. is led at a const. velocity thruh a Sn or Cu tube 18 cm. long and 0.22 cm. in diam., and immersed in liquid 0, only 0.2-0.7% of the Em. escapes, but if the tube be made of glass 60-70% escapes. The poor heat conductivity of the glass does not appear to be the cause of this difference, for the condensation in a glass tube is increased if a small Cu wire be placed inside it. Nearly as good condensation can be obtained in a glass tube as in a metal one if the tube is long enough. A glass tube 1.6 m. long gives the same results as one of Sn 18 cm. long.

Experiments to Determin the Period of Ionium. F. Soddw, 7, 295-300.—The author discusses the difficulties encountered in detg. the period of ionium, and gives data which lead him to the value 132,000 years as the mean period of life of ionium. This is obtained from a knowledge of the fraction of the equil. amt. of Ra in autunite and the age of the mineral estd. from its He content.

C. H. V.

Chemical Investigation of the Residues from the Preparation of Radium which Contain Actinium. I. C. Auer v. Welsbach. Monatsh., 31, 1159-1202; Z. anorg. Chem., 69, 353-91.—This article is too full of details for a brief abstract. 1800 kgm. of the hydrates obtained by the pptn. with NH, of the HCl exts. (Debierne process for the prep. of Ra) from 10 tons of pitch blende residues, were given to the author, by the Radium Commission of the Imperial Academy of Sciences, to det. the chem. nature of the radioactive substances contained, and if possible to isolate these. A qual. exam. showed the main constituents to be iron, alumina, lime, rare earths, bismuth, uranium, silica and many other elements in smaller quantities. Two general methods were used. The "sulfate" method, which while complicated gave more insight into the chem. relations, and permitted the prep. of radioactive products in part without the use of ignitions. The "oxalate" method was worked out on the basis of results obtained in working up 120 kg. of the paste by the "sulfate" method. Aside from polonium (Ra F) there was present ionium and actinium. The former seemed homogeneous whereas the latter seemed complex. Ionium follows the reactions of thorium, while actinium comes between lanthanum and calcium. Thorium salts containing about  $2^{1}/2\%$  of ionium were prepared—data as to the content of actinium preps. are not yet available. The author says that many observations lead him to believe that ionium can cause chemically related substances to become radioactive on longcontinued action. C. H. VIOL.

Radioactive Equilibrium in Cotunnite (Rossi). 8.

System Ethyl Alcohol, Acetic Acid, Ethyl Acetate and Water (JONES, LAP-WORTH). 10.

### 4. ELECTROCHEMISTRY.

C. G. FINK.

The Electro Steel Works of the Eicher Hüttenverein LeGallais, Metz & Co. E. Bian. Stahl w. Eisen, 31, 217-24.—A detailed description of the equipment of the above works is given. A chem., a physical and a microscopical laboratory are maintained. Results of expts. given show that electro steel is comparatively resistive to the action of acids.

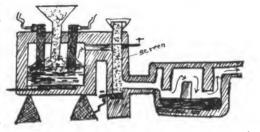
E. J. Crane.

Electric Steel Processes as Competitors of the Bessemer and Open Hearth. A. E. Green. Trans. Am. Electrochem. Soc., 19, 233.—A long discussion of the relative merits of these two forms of furnaces as regards the removal of impurities. Special mention is made of a method of blowing in a mixture of CO and CO, so that an atmosphere is maintained which is reducing toward Fe and oxidizing toward C.

W. E. RUDER.

The Johnson Electric Zinc Furnace. J. W. RICHARDS. Trans. Am. Electrochem. Soc., 19, 311.—A small 25 kw. furnace of this type has operated quite successfully, producing Zn at the rate of about 2 tons per h.-p.-yr. The harmful effect of CO<sub>2</sub> in pro-

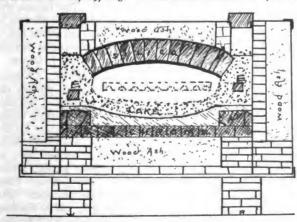
ducing a large % of blue powder is overcome by passing the vapors from the furnace thru a hot carbonaceous screen and reducing the CO<sub>3</sub> to CO. The slag loss is not over 0.5% Zn. The temp. is held so that the gases pass into the condenser at about 850°. The working temp. of the slag body was 1250° to 1300° and the smelting



cap. per cu. ft. of smelting zone is 50 times as rapid as in the fire clay Zn retort. A larger furnace is being constructed. The furnace is continuous and is of the buried arc type.

W. E. RUDER.

An Electric Furnace for Heating Bars and Billets. T. F. BAILY. Trans. Am. Electrochem. Soc., 19, 285.—A resistance furnace of special construction for heating



bars for forging, etc., is described. The resister is of crushed coke placed at the bottom of the heating chamber and the electrodes imbedded in it in such a way that the heating at electrodes is very small and the hottest part of the resister is that nearest the charge. The lining is of chrome and silica bricks and the insulation of wood ash (see sketch). The bottom lining has to be re-

newed about once a week. The principal advantages of the elec. furnace are non-oxidizing atm., freedom from soot, and high thermal efficiency, which is given as 33

to 65%, depending on the size of the furnace, as against 4% for oil fired furnaces. Diagrams and cuts of the furnace are given.

W. E. RUDER.

The Condensation of Zinc Vapor from Electric Furnaces. F. T. SNYDER. Trans. Am. Electrochem. Soc., 19, 317; Chem. Eng., 13, 209.—The author, in a detailed consideration of the problem of the condensation points out the losses due to poor design and describes the condenser used on the Lynen furnace (Metallurgy of Zn, Ingalls p. 486) and points out its deficiency in the time it provides for diffusion. The condenser has diffusing capacity for only 1/2 of the Zn vapor for which it was designed.

W. E. RUDER.

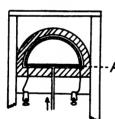
Action of Glacial Phosphoric Acid on Various Alloys Obtained in the Electric Furnace. M. WUNDER AND B. JANNERET. Compt. rend., 152, 1770-1.—The following elements and alloys when heated with excess of glacial H<sub>2</sub>PO<sub>4</sub> at 230° dissolve to a clear liquid, sol. in H<sub>2</sub>O and HCl: Zr, W, Fe-Si, Fe-Ti, Fe-Zr, Fe-Va, Si-Mn, Ti-N, Fe-Si-Al, B-Ni; Si and carborundum dissolve partially with formation of a white ppt., insol. in H<sub>2</sub>O and HCl.

G. R. FONDA.

Observations on Laboratory Production of Aluminium. H. K. RICHARDSON. Electrochem. Eng. Lab., Penn. State Col. Trans. Am. Electrochem. Soc., 19, 159-70.—Satisfactory runs were made by students using a specially designed app. (construction described) obtaining an amp. efficiency of from 60-70%, electrolyzing a mixture of 85% natural cryolite and 15% Al<sub>2</sub>O<sub>3</sub> with a c. d. of approx. 3 amps. per sq. cm. of anode surface. The anode (carbon) effect appears to consist of 2 kinds: (a) a gaseous envelope which opens the circuit; and (b) non-melting of the electrode causing numerous small arcs to form from electrode to electrolyte. Prof. J. W. Richards stated in discussion his belief that the failure to realize higher efficiency in the process was due to soln. of Al in the electrolyte.

C. N. Moore.

An Electric Resistance Furnace with a Resister of Base Metals. H. SIEBERT.



Berlin. Chem. Zig., 35, 443.—The author briefly describes an elec. furnace the resister of which is made of base metals, (Fe, Ni and their alloys of high m. p.). The hollow airtight space A (see fig.) is supplied with an inlet and outlet tube so that an indifferent or reducing gas may be passed thru, such as H which prevents oxidation. This furnace will withstand greater mechanical strain and is as efficient in other ways as one using a Pt resister, and the initial cost is much less. It can be used with direct or alternating current.

E. J. CRANE.

The Laszczynski Electrolytic Copper Process. WALTHER STOEGER. Chemists, 2, 129-31, 147-8.—Compared with the Siemens and with the Hoepfner process the new one is very simple. The objectionable influence of iron salts present in the electrolyte is entirely overcome by encasing the Pb plate (insol.) anodes in tightly fitting heavy cotton bags; even when large quantities of Fe are present, good bright Cu deposits are obtained. The process is in successful operation at the Miedzianka Mine, near the German-Austrian border. The ore, largely sulfide, ranges between 15% to 40% Cu. To ensure complete oxidation to sulfate, the ore is finely ground, mixed with a binder and pressed into briquets; after drying, these are very porous. They are dried and roasted in a specially designed furnace which is fully described. The roasted briquets are pulverized and leached in a soln. of 7% H<sub>2</sub>SO<sub>4</sub>. After leaching the soln. contains 5% Cu and 1% free H<sub>2</sub>SO<sub>4</sub> and is ready for electrolysis. The electrolyzing tanks are lead-lined. There are 9 Pb anodes and 8 thin sheet Cu cathodes. Wooden stirrers are placed between the electrodes. Each tank takes 900 amp. and

2.25-2.50 volts. The quantities of Cu deposited correspond very closely to the current equivalent. The cotton anode bags need to be renewed only once or twice a year. The stirring of the bath does not disturb the electrolyte enclosed in the cotton pores: upon this principal the whole process is based and the absence of injurious effects of the Fe salts on the Cu accounted for.

C. G. Fink.

The Action of a Third Disconnected Metal in Electrolytic Baths. Anon. Electrochem. Z., 18, 113.—It is shown that a metal-lined electrolyzing tank or an uncoated metal tank always carries a positive charge on the lining and should this be sol. or is in contact with any metal sol. as anode the electrolyte is quickly contaminated. For example, a Ni plated Fe object that falls to the bottom of the container is rapidly dissolved owing to the fact that both Ni and Fe are positive to the Pb lining. If however the object be Cu the action is very slight since the Cu is electro-negative to Pb.

W. E. RUDER.

Thermodynamics of Concentration Cells. H. S. CARHART. J. Wash. Acad. Sci., 1, 20–1.—An experimental investigation of the e. m. f. of various conc. cells with amalgams of different conc. as the electrodes. In every case a linear relation is established between the e. m. f. and temp. The expression for E has the form  $E = E_k + aT$ , where  $E_k$  is the e. m. f. due to the internal change of energy H. The coeff. a is not zero as Nernst assumed it to be. For the zinc amalgams used, the equation expressing the e. m. f. is E = -0.001455 + 0.00003084T. Electrolytic thermoelectromotive forces were measured also and showed a linear relation over a range of temps. as much as 25 or 30°. Two calomel cells were constructed, one with a positive temp. coeff. and the other with an equal negative one. The former is set up with a 10% Zn amalgam, and the latter with one of about 0.8%. When the two cells are joined in series, the sum of their electromotive forces is independent of temp. thru at least the range 8 to 34°.

Selenium. G. L. ADDENBROOKE. Electrician, 67, 591-2.—A review of a number of researches on the physical properties of Se, more particularly its sp. resistance and sp. inductive capacity.

C. N. Moore.

The Electrical Precipitation of Suspended Particles. F. G. COTTRELL. J. Ind. Eng. Chem., 3, 242-50.—A description of the industrial development and application of a process based on the migration of charged particles under the influence of an electical. High potential d. c. is used as a source of energy, and asbestos or mica as discharge points. The application to gases from a H<sub>2</sub>SO<sub>4</sub> contact plant, Pb blast furnace, pyrites and mat roasters, parting house furnes, and Cu smelters is described.

GEORGE W. MOREY.

Neon Luminescent Lamp. G. CLAUDE. Ind. elec.; thru Elec. World, 58, 456.

—The author finds that the lowering of the pressure in the Moore tubes during life is due chiefly to a vaporization of the electrodes. Interesting data are given of deposits resulting therefrom and the discovery of He in exhausted tubes. The vaporization of the electrodes can be practically overcome by constructing them with very large surfaces.

L. K. MULLER.

Selective Radiation from Various Substances. III. W. W. COBLENTZ. Bull. Bw. Stand., 7, 243-94.—In the present investigation of the  $C_2H_2$  flame, the object was an exact determination of emissivity and absorptivity in the visible part of the spectrum. The expts. developed that the earlier spectrophotometric data had been misinterpreted. A rapid increase in the transparency of the flame is shown in the region from 0.7  $\mu$  to 0.8  $\mu$ . The ratios obtained by a comparison of the emissivities of the  $C_2H_2$  flame with those of the Nernst glower and C filament prove that a band of selective absorption extends thruout the visible spectrum with a max. at about

0.6  $\mu$ , and that the absorption becomes more uniform beyond 0.75  $\mu$ . According to expts. on the Welsbach mantle, a Nernst glower made of the same material must be heated to a much higher temp. to attain a luminous effect comparable with that of the gas mantle. The high light emissivity can be accounted for on a purely thermal basis by considering the physical condition of the Ce oxide at high temp. where there seems to be a broadening of the adsorption band toward the red. In a color or spectrophotometric match between a Nernst glower and a W filament, there is no true superposition of the spectral energy curves in the visible spectrum; they intersect however at an extremely small angle, giving an apparent superposition under the sensibility of the eye.

G. R. Fonda.

Continuous Diminution in Ultraviolet Radiation from Quartz Mercury Vapor Lamps at High Temperature. J. Courmont and C. Nogier. Compt. rend., 152, 1746-7.—The continuous spectrum characterizing lamps run at high temp. becomes much less intense with increasing age of the lamp. Similar decreases during life in the emission of ultraviolet light can be observed by the diminished intensity of photochemical and biological actions. This may be due to a grayish deposit on the interior walls of the lamp or to a change in the composition of the vapor. It may be partially avoided by cooling the quartz walls.

G. R. Fonda.

Comparison of Illuminants. S. W. ASHE. Trans. Illumin. Eng. Soc., 6, 503-17.

—The author claims that the only fair basis of comparison is the av. candle power performance during life. The values given are averages in % of the initial c. p. Gas mantle (high grade), 70; Hg vapor tube (a. c.), 75; Hg vapor tube (d. c.), 61; enclosed C arc, 78; magnetite arc (6.6 amp), 90; drawn wire W lamp (Mazda), 94. The value 94 is for 60 and 100 watt (100 volt) lamps after 1000 hrs. at 1.18 w. p. c.

L. K. MULLER.

Ozone and its Applications. Anon. Ind. chim., 11, 121-3.—Review.

Chas. A. Rouiller.

Ger., 235,666, Oct. 26, 1909. E. ACHENBACH, Wilhelmsburg a/Elbe bei Hamburg. In carbonizing, in the open air, the binding agent in elec. filaments produced according to the paste process, the oxidizable metal powder forming the ground substance of the filament is provided with a coating of Ag in the known manner.

Ger., 235,706, June 5, 1910. CENTRALSTELLE FÜR WISS. UNTERS., Neubabelsberg. In the electrolytic production of halogen-oxygen compounds, compds. of the rare earths, in acid soln., are added to the electrolyte. The addition of Ce-, Er-, La-, or Yt-chloride renders possible a high current yield of NaClO<sub>3</sub>.

Ger., 236,002, Jan. 16, 1908. SIEMENS & HALSKE, Berlin. App. for the electrolysis of aqueous salt solutions such as ZnSO<sub>4</sub>, Cu(NO<sub>2</sub>)<sub>2</sub>, NaCl solns., or the like, whereby also free acid can be present. As anodes, massive, artificially prepared MnO<sub>2</sub> is employed. The cryst. MnO<sub>2</sub> conducts the current well and may be readily formed into sticks or plates.

#### 5. PHOTOGRAPHY.

W. B. HOLMES.

Studies on the Photo-haloids. W. REINDERS. Delft. Z. physik. Chem., 77, 213-26; cf. C. A., 5, 2223, 2372.

Colloid Chemistry and Photography. LUPPO-CRAMER. Frankfurt. Z. Chem. Ind. Kolloide, 8, 240-5.—Yellow Ag sol has the smallest, blue the largest, particles.

The smallest particles (seeded with a large volume of colloidal Ag soln. (Idem., C. A., 5, 637)), give first a red sol, soon changing to green if formed in purified gelatin soln. The same procedure with unpurified gelatin gives a yellow-red sol. By washing, constituents must be removed from the gelatin which hinder the change from red to green. probably NaCl; by adding NaCl to washed gelatin the same effect as with unwashed gelatin is obtained. The change of the red particles to green is probably an agglutination of a different sort from that which gives the blue particles, for the green sol may be changed back to red by small quantities of haloid salts as NaCl, KBr, KI, or KCN, while blue and violet remain unchanged. Five samples of 100 cc. 0.4% AgNO, soln. were seeded with a 0.05% colloidal Ag soln. prepared according to Carey Lea and containing small amts. of dextrin as follows: (a) 20 cc., (b) 10 cc., (c) 5 cc., (d) 2 cc., (e) 0 cc. These were then reduced with hydroquinone and the color of the colloid from (a) to (e) went from black to gray, and the speed of bleaching of the colloid by HgCl, decreased from (a) to (e), (a) bleaching in  $2^{1}/2$  min. and (e) in 24 hrs. If the reduction took place in the presence of NaCl the time of bleaching of the sol formed with only small amts. of colloidal Ag or none, was unchanged, but sample (a) seeded with a large amt. of colloidal Ag bleached in 1.5 hours without NaCl, and in 3 to 5 min. in the presence of NaCl. The catalytic action of such a Ag sol on H<sub>2</sub>O<sub>2</sub> can be almost entirely prevented by gelatin, agar, albumin or dextrin and in the presence of such a colloidator the H<sub>2</sub>O<sub>2</sub> causes the soln. of the Ag sol. The speed of soln. of the Ag particles is independent of the degree of dispersion in the absence of chlorides, etc., but in their presence the rate of soln. is decreased and decreased more the greater the degree of dispersion of the colloid, i. e., the greater the degree of dispersion of the Ag, the more readily is it halogenized. If a negative is treated with a halogen-containing oxidizing agent the faintly illuminated parts that contain the more dispersed Ag become halogenized on the surface, which then prevents the Ag from being attacked by the oxidizing agent. The coarsest Ag particles first dissolve and are attacked by the haloid only after getting outside the gelatin layer. The same difference is observed when HNO, and HgCl, act on colloidal and gray Ag; the colloidal is changed instantaneously to a mixture of AgCl and HgCl, while the gray is dissolved by the HNO, and then changed into AgCl, no HgCl being formed. Such a mixture of HNO<sub>2</sub> and HgCl<sub>2</sub> causes a bleaching of the high lights and strengthening of the shadows in a negative. H. ISHAM.

Gradation of Silver Bromide Gelatin Layers in the Ultraviolet. ERICH STENGER. Photochem. Lab. kgl. techn. Hochschule, Charlottenburg. Physik. Z., 12, 580–2.— Unsubdued white light, ultraviolet and orange-red portions of the spectrum were compared in their effect on AgBr gelatin plates. The filters used consisted of equal pts. of nitrosodimethlyaniline (1:5000) and rhodulin violet (1:5000) for the ultraviolet and equal pts. of eosin (1:2000), uranin (1:2000), and tartrazine (1:100) for the orange-red portion of the spectrum. The first filter is transparent for rays of wave length 300–385  $\mu\mu$  and the second for those wave lengths from the D line to near the red end of the visible spectrum. Measurements of the darkening of the plate were made with a light Marten polarization photometer. The data, graphically expressed, show that gradation in the ultraviolet agrees with that in the visible spectrum, and that therefore the gradation in the invisible short wave spectral region is independent of wave length.

Time Development. C. E. Kenneth Mees. Am. Photo., 5, 212-9.—Development is considered chemically by the reaction of AgBr with an alk. soln. of hydroquinone; physically by the structure of jellies and rates of diffusion of solns. into jellies; and physico-chemically by the removal of Ag from the soln. As AgBr reacts with the alk. hydroquinone, Ag accumulates so that the soln. is satd. with respect to Ag and the reaction stops unless the Ag be removed. An unexposed plate will develop

because the Ag is in the condition of a solute in a supersatd. soln. Exposing the plate furnishes a nucleus for the deposition of Ag, analogous to the addition of a minute crystal of the solute to a supersatd. soln. The time for which a plate must be developed to obtain a given degree of contrast depends upon 3 factors: maximum contrast attainable, velocity const., and temp. coeff. The max. contrast given by a plate is reasonably const. from batch to batch. Sudden changes in weather or water supply, while making the emulsion, may cause variations; also any change in the gelatin used. The velocity const. of development at the same temp. and for the same degree of development varies greatly with different batches of the same plate. This is mainly governed by the rate at which plates dry, which, even in artificial drying system, is affected by external conditions. Tables of corrections for variations in temp. of developer from a standard are misleading. The time must be found by trial with each batch of plates.

L. W. Riggs.

Comparative Studies on Intensifiers and Reducers. E. STENGER. Z. Reprodukt., 13, 84-7; see C. A., 5, 636, 1878, 2225.—Persulfate preps. which show no decomp. and react only very weakly acid have very little reducing power. The addition of HNO<sub>3</sub> to freshly prepared solns. increases this power, but the addition of HNO<sub>3</sub> to solns. which are acid in reaction (decomposed) does not increase the power proportionally. HCl acts like NaCl causing a protection from reduction due to the formation of AgCl. NH<sub>2</sub> or KCN retard the reduction and in water containing Cl destroy the protective effect of the AgCl. KOH also retards the reduction but does not destroy the protective effect of the AgCl in water containing Cl. Persulfate solns. in hard water do not cause as much reduction as those in soft water. The reduction is independent of the kind of plate.

GEO. W. STRATTON.

Comparative Studies on Intensifiers and Reducers. E. STENGER. Z. Reprodukt., 13, 100–105; see C. A., 5, 636, 1878, 2225—Plates developed with either Amidol or Rodinal show a similar reduction with the persulfate reducer when used in distilled or tap-water. The action is more rapid with the Amidol plates. Films are reduced a small, but measurable, amt. Because the Cl content of tap water varies considerably the author suggests that persulfate soln. be made with distilled water. He gives the following proportions: As a progressive reducer, 1 g.  $(NH_4)_2(SO_4)_2$ , 100 cc. distilled water; as a persulfate-like reducer, 2 g.  $(NH_4)_2(SO_4)_2$ , 100 cc. distilled water, 1.5–2.5 cc. NaCl. The paper contains a summary of the author's previous work on this subject.

The Theory and Practice of Intensifying. A. SCHULLER. Phot. Rundschau, 25, 141-4.—The author describes a method of measuring and representing in the form of curves the amount of intensification of photographic plates.

GEO. W. S.

Physical Development of a Radiographic Image on a Plate which has been Fixed with Sodium Thiosulfate and Thoroughly Washed. M. Chanoz. Compt. rend., 152, 1576-7.—Plates exposed to X-rays and then developed before and after fixing are compared. The opacity of the plate developed after fixing increases within certain limits according to length of time the developer acts and is greater than with the plate developed in the ordinary way. The minimum exposure necessary to produce a visible image is 10 times as great in the case of the plate developed after fixing as with the plate developed in the usual way.

W. B. Holmes.

Application of the Chemical Harmonica to Chronophotography. L. Benoist. Compt. rend., 153, 96-7.—The author applies the musical flame to chronophotography and gives the requisit conditions for securing stable vibrations, and for producing flames making 200-1000 vibrations per sec.

George W. Morey.

Diaminophenol as a Developer in Tropical Countries. A. AND L. LUMIÈRE AND

A. Sevewetz. Photo-Era, 27, 15-6.—The object of this research was to provide a developer capable of being used at 100-104° F without alteration or softening of the gelatin film and without fogging the plates. Metoquinone, metol-hydroquinone, pyro and diaminophenol were tried with results favoring the following formula: Diaminophenol 5 g., Na<sub>2</sub>SO<sub>4</sub> 30 g., cryst. (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> 250 g., KBr 3 g., water 1000 cc. In place of the 250 g. (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> 150 g. Na<sub>2</sub>SO<sub>4</sub> may be used. This developer gives excellent negatives in 3 min. of development, and is recommended whenever great keeping power is not essential.

L. W. RIGGS.

Treatment of Poisoning from Photographic Chemicals. PAUL L. ANDERSON. Am. Photo., 5, 458-62.—Rules to be observed in the dark room are given, and the usual treatment for poisons except that carbonates are recommended as antidotes to mineral acids.

L. W. RIGGS.

Profitable Saving of Photographic Waste. A. J. JARMAN. Am. Photo., 5, 356-8.

—Thirty million oz. of Ag are consumed yearly in the U. S. in photography. In the photo. lab. of the Sci. Am. the waste Ag was pptd. from hypo soln. by strips of Fe hoop suspended in a 5-gal. stone-ware jar. The Ag ppt. was washed, collected, and refined. Amt. recovered in one year 26.12 oz. worth \$14.36. Cost of refining \$2.15. Pptn. by Fe is preferred to that by K<sub>2</sub>S or Zn which cause the evolution of H<sub>2</sub>S. From the Au toning bath, 130 grains of AuCl<sub>2</sub> were recovered.

L. W. RIGGS.

## 6. INORGANIC CHEMISTRY.

#### H. I. SCHLESINGER.

Producing Ozone by a Chemical Reaction. P. MALAQUIN. J. pharm. chim., 3, 329-35.—Place 20 g. NH<sub>4</sub> persulfate in 15 g. pure HNO<sub>3</sub> of 36 Bé. in a thin glass vessel appropriately provided with glass connections. Wash out the air with CO<sub>4</sub>, heat to 70-75° and wash the resulting gas with about 20 cc. of 20% soln. of KOH. The gas consists of 94-95% O, 3-4% ozone and 4-4.5% N.

V. K. Chesnut.

Catalytic Action of Copper Oxide. J. STRACHAN. Chem. Lab., Ballyclare Co., Antrim. Chem. News, 103, 241-2; cf. Meuner, C. A., 5, 1545; Sabatier, C. A., 2, 2687, 3057, 3058; 3, 2675; 4, 2094, 2098.—A warm, slightly oxidized, brass wire-gauze catalyzes the oxidation of MeOH in a mixt. of MeOH and air as it issues from a Berthol spirit Bunsen, aldehyde being produced. Finely divided CuO, deposited on asbestos by dipping the latter into conc. CuSO<sub>4</sub> soln. and then heating, will when warm, catalyze the oxidation of the gases issuing from a Bunsen burner. The asbestos becomes red hot and may ignite the gases. The copperized asbestos will also catalyze the oxidation of H in air, ignite an explosive mixture of H and O and catalyze the oxidation of MeOH vapor, when mixed with air while coming from a flask of boiling MeOH. Dentritic growths of CuO in paper cause oxidation of the cellulose. For some of the above expts. FeCl<sub>8</sub> may take the place of CuSO<sub>4</sub>. The catalytic action of oxides may be a source of danger in the miner's safety lamp.

Some Properties of Potassium Nitrite. E. Donath. Chem. 2tg., 35, 773-4.—Of the two layers (Fischer, Ann. Phys. Chem., (1849) 14, 115)) obtained by mixing an equal vol., or more, of 96% EtOH with a nearly satd. aq. soln. of KNO<sub>2</sub>, the lower contained 71.9% of KNO<sub>2</sub>, the upper, alcoholic, 6.9%. KNO<sub>2</sub> is then capable of sepg. aq. alc. into a less alcoholic layer. With MeOH there is no sepn. of the soln. into layers, but a heavy pptn. of pure KNO<sub>2</sub>. Since KOH is sol. in MeOH, the latter may be used to sep. the former from KNO<sub>2</sub>. The turbidity produced when a conc. soln. of KOH is mixed with MeOH is due to the sepn. of salts. Conc. aq. solns. of NaOH are pptd.

by MeOH, while NaNO, solns. are not; neither is there a sepn. into two layers by the addition of EtOH (cf. Fehling's Handwörterbuch der Chem.).

D. M. LICHTY.

Extraction of Gas from Copper Heated in a Vacuum. M. Guichard. Compt. rend., 153, 104-7.—Cu, heated in a vacuum to about 600°, emits gas in quantities at first easily noticeable, later only by means of a Macleod gage. If the surface of the Cu is small, the evolution of gas becomes insensible after a time, but again becomes measurable if the Cu is left at ordinary temp. for some hrs. and is then again heated. The gas in the interior of the Cu evidently reaches the surface by very slow diffusion. A given wt. of Cu of large surface gives out more gas in a given time than the same wt. of Cu of small surface. At the m. p. of Cu the evolution of the gas was slow, due to their higher solubility at higher temps. Reduction of the pressure exerted on the melted Cu did not facilitate the release of the gas as much as might be expected, because the solubility of the gas varies only as the sq. root of the pressure (Sievarts, Z. Electrochem., 16, 707). The gas was generally a mixture of CO<sub>2</sub>, CO, N and H, in variable amts.

Some Reducing Actions of Mercury. D. BORAR. Univ. Coll., Cardiff. J. Chem. Soc., 99, 1414-6; Proc. Chem. Soc., 27, 128.—Mercury with KMnO<sub>4</sub> soln. gives rise to KOH, MnO<sub>2</sub> and HgO (cf. Kirchman, Arch. Pharm., 200, 203; Giles, Chem. News, 15, 1204); with K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in HCl soln., to CrCl<sub>2</sub> and Hg<sub>2</sub>Cl<sub>3</sub>; with ferric salts in HCl soln., to ferrous salts and Hg<sub>2</sub>Cl<sub>2</sub>; K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> soln., to K<sub>2</sub>SO<sub>4</sub> and a basic mercuric sulfate; with CuSO<sub>4</sub> in HCl soln., to CuCl and Hg<sub>2</sub>Cl<sub>2</sub>; with C<sub>2</sub>H<sub>3</sub>NO<sub>2</sub> in HCl soln., to a little C<sub>4</sub>H<sub>3</sub>NH<sub>3</sub>. With KNO<sub>2</sub>, KClO<sub>3</sub> and Na<sub>2</sub>O<sub>3</sub> solns. no action occurred. Hg may be used for reducing ferric compds. in the estn. of Fe, the reduction proceeding rapidly and completely at 60 to 70° (cf. Carnegie, J. Chem. Soc., 53, 471). D. M. LICHTY.

Potassium Ammono-plumbite. E. C. Franklin. Stanford Univ. J. Phys. Chem., 15, 509-20.—Potassium ammono-plumbite, PbNK.2.5NH<sub>2</sub>, was obtained by the interaction of KNH<sub>2</sub> and PbNH, both dissolved in liq. NH<sub>3</sub>. The PbNH was prepared by the action of KNH<sub>2</sub> on PbI<sub>2</sub>, KI and NH<sub>3</sub> being formed at the same time. From liq. NH<sub>3</sub>, the PbNK.2.5NH<sub>3</sub> seps. in colorless crystals, which readily give off successively 0.5 and one mol. of NH<sub>3</sub>.

D. M. LICHTY.

The Monohydrate of Barium Chloride. A. KIRSCHNER. Kopenhagen. Z. physik. Chem., 76, 174-8.—BaCl<sub>2</sub>.2H<sub>2</sub>O is dissolved in MeOH and the soln. rapidly filtered. From this, on standing, BaCl<sub>2</sub>.H<sub>2</sub>O separates in rhombic crysts. It is less sol. in dil. alc. than in abs., hence addition of the small amts. of H<sub>2</sub>O hastens the pptn.

H. I. S.

Pyridine Compounds of Tin Halides. P. PFEIFFER, et al. Chem. Lab., Univ. Zurich. Z. anorg. Chem., 71, 97-120.—When SnCl<sub>4</sub> is added to Py, both being dry and cold, tin tetracklorodipyridine, SnCl<sub>4</sub>Py<sub>2</sub>, separates, and tin tetrabromodipyridine, SnBr<sub>4</sub>Py<sub>2</sub>, is formed in the same manner. Both are white powders, insol. in ordinary solvents, and decompose on heating. When SnI<sub>4</sub> is placed in a Py atmosphere over soda-lime, Py is taken up until the mass becomes liquid. A definit compound, SnI<sub>4</sub>-5Py, is probably formed. Tin methyltrichlorodipyridine, SnMeCl<sub>2</sub>Py<sub>2</sub>, formed by adding Py to either an Et<sub>2</sub>O soln. of SnMeCl<sub>2</sub> or to solid SnMeCl<sub>3</sub> and warming; colorless crystals from Py, sol. in cold, easily in hot Py and EtOH, insol. in Et<sub>2</sub>O, easily sol. in H<sub>2</sub>O. Tin methyltribromodipyridine, SnMeBr<sub>3</sub>Py<sub>3</sub>, is an amorph. white powder, becoming yellow on heating and melting to a cloudy liquid at 203°, difficultly sol. in cold, easily in hot EtOH and Py, easily sol. in H<sub>2</sub>O. When SnMeI<sub>3</sub> is placed in a Py atmosphere with Na<sub>2</sub>CO<sub>3</sub> it takes up Py until it liquefies. The liquid mass loses Py over CaCl<sub>2</sub>, forming the compound, SnMeI<sub>3</sub> + 4Py, yellow powder, decomp. on heating, sol. in H<sub>2</sub>O and Et<sub>2</sub>O, insol. in ligroin. Tin dimethyldichlorodipyridine, SnMe<sub>2</sub>Cl<sub>2</sub>Py<sub>3</sub>.

from SnMe<sub>2</sub>Cl<sub>2</sub> and Py, glistening colorless crystals, m. 163°, sol. EtOH, H<sub>2</sub>O, difficultly sol. in ligroin. Tin dimethyldibromodipyridine, SnMe<sub>2</sub>Br<sub>2</sub>Py<sub>2</sub>, forms glistening crystals, m. 172°, easily sol. in H<sub>2</sub>O and hot EtOH, difficultly sol. in ligroin. Tin dimethyldibiododipyridine, SnMe<sub>2</sub>I<sub>2</sub>Py<sub>2</sub>, from SnMe<sub>2</sub>I<sub>2</sub> and Py, small colorless crystals, which become yellow in the light and m. at 147° (decomp.). Easily sol. in EtOH. Tin dipropyldichlorodipyridine, SnPr<sub>2</sub>Cl<sub>2</sub>Py<sub>3</sub>, from SnPr<sub>2</sub>Cl<sub>2</sub> and Py, forms glittering colorless needles, m. 114°, sol. in C<sub>6</sub>H<sub>6</sub>, EtOH, CHCl<sub>2</sub>, (CH<sub>3</sub>)<sub>2</sub>CO, and Py. Tin dipropyldibromodipyridine, SnPr<sub>2</sub>Br<sub>2</sub>Py<sub>2</sub>, colorless glittering needles, m. 128°, decomp. in air, sol. in C<sub>6</sub>H<sub>6</sub>, EtOH, CHCl<sub>2</sub>, (CH<sub>3</sub>)<sub>2</sub>CO, and Py. Tin dibutyldichlorodipyridine, SnBu<sub>2</sub>Cl<sub>2</sub>Py<sub>2</sub>, from SnBu<sub>2</sub>Cl<sub>2</sub> and Py, colorless prisms, m. 65–66°, sol. in Me<sub>2</sub>CO, Et<sub>2</sub>O and EtOH. When a soln. of SnBu<sub>2</sub>Cl<sub>2</sub> in much Py is allowed to cryst. in a CaCl<sub>2</sub> desiccator the product is a

basic compound, Bu<sub>2</sub>Sn Cl

Tin dibutyldibromodipyridine, SnBu<sub>2</sub>Br<sub>2</sub>Py<sub>2</sub>, crystals,

m. 77-78°, easily sol. in Et<sub>2</sub>O and EtOH, difficultly sol. in ligroin. When SnPh<sub>2</sub>Cl<sub>2</sub> is warmed with excess Py, the compound SnPh<sub>2</sub>Cl<sub>2</sub>Py<sub>2</sub> + 2Py seps. in glittering colorless tablets, m. 155-6°, sol. in hot Py, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>2</sub>, Me<sub>2</sub>CO, and EtOH. In the air or in vacuum this compd. loses Py, forming tin diphenyldichlorodipyridine, SnPh<sub>2</sub>Cl<sub>2</sub>Py<sub>2</sub>, better obtained by crystn. of the above compd. from abs. EtOH; glittering colorless needles, m. 151°. With SnPh<sub>2</sub>Br<sub>2</sub> and Py the compound SnPh<sub>2</sub>Br<sub>2</sub>Py<sub>2</sub> + 2Py, glittering colorless tablets, m. 160°, sol. in Py, EtOH, Me<sub>2</sub>CO, C<sub>2</sub>H<sub>4</sub>, CHCl<sub>2</sub>. From this compd., tin diphenyldibromodipyridine, SnPh,Br,Py,, is obtained as above, glittering colorless needles, m. 165°, same solubility as above compd. When ditolyl-p-tin bromide, (C<sub>8</sub>H<sub>4</sub>.-CH<sub>2</sub>)<sub>2</sub>SnBr<sub>2</sub>, prepd. from (C<sub>5</sub>H<sub>4</sub>.CH<sub>2</sub>)<sub>4</sub>Sn and Br, is dissolved in hot Py and the soln. cooled, tin ditolyldibromodipyridine, Sn(C<sub>2</sub>H<sub>4</sub>.CH<sub>2</sub>)<sub>2</sub>Br<sub>2</sub>Py<sub>2</sub>, colorless crystals, m. 172-6°, is obtained. Tin triphenylchlorodipyridine, SnPh,ClPy, from SnPh,Cl and Py, forms colorless crystals, m. 81-4°, sol. in C<sub>8</sub>H<sub>6</sub>, Py, EtOH, Et<sub>2</sub>O, and ligroin. Tin triphenylbromodipyridine, from SnPh<sub>2</sub>Br and Py, forms colorless crystals, m. 78-84°, sol. in Py, C<sub>e</sub>H<sub>e</sub>, Et<sub>2</sub>O, EtOH, ligroin. The 2 latter compds. lose all their Py in the air or in vacuum. GEORGE W. MOREY.

Thorium Arsenates. G. A. BARBIERI. Atti accad. Lincei, 19, II, 642-5; Chem. Zentr., 1911, I, 795.—Boiling Th(NO<sub>4</sub>)<sub>4</sub> soln., treated with 4 mols. H<sub>2</sub>AsO<sub>4</sub> (40%), gives the compound Th(AsO<sub>4</sub>H)<sub>2</sub>.6H<sub>2</sub>O, cryst. powder consisting of minute cubes, insol. in H<sub>2</sub>O. With 8 mols. H<sub>2</sub>AsO<sub>4</sub> is obtained the compound Th(AsO<sub>4</sub>H<sub>2</sub>)<sub>4</sub>.4H<sub>2</sub>O, slender needles, gives the first salt when treated with H<sub>2</sub>O. Chas. A. ROUILLER.

Silicon Sulfides. II. LIVIO CAMBI. Lab. elet.-chim. r. ist. tecn. sup., Milano. Atti accad. Lincei, 20, I, 433-40; cf. C. A., 5, 1567.—The white compd. resulting from the hydrolysis of yellow SiS has the comp. H<sub>2</sub>Si<sub>2</sub>O<sub>3</sub> of silicoformic anhydride. It is easily oxidized in contact with H<sub>2</sub>O at room temp., evolving H, quite sensitive to alk. and NH, and to HF, reduces mercuric to mercurous salts and Hg; heated in vacuo to 400°, it decomp. into H, Si and SiO<sub>2</sub>. With dry HCl at 240-60°, the yellow SiS gives H<sub>2</sub>S and SiHCl<sub>2</sub>. The reddish yellow product of hydrolysis of black SiS, on treatment with dry HF, increases in Si content to 96% and in d<sup>15</sup> to 2.082, but retains all the other properties of the original compd.; in reactivity it corresponds to the active amorphous Si described by Berzelius. Treated at 290-300° with dry HCl, black SiS gives H, H<sub>2</sub>S, about 3% of dark red Si and a liquid, 30% of which consists of SiHCl and 70% of SiCl, and SiCl, SH. The black sulfide must therefore be a mixture of Si and SiCl2, with possibly SiS and intermediate compds. That there is a reversible transformation of SiS into Si + SiS, was shown by the following expts. When, in the sublimation of the crude sulfide a cooling tube was interposed, the first layers which were deposited were orange-yellow and darkened to reddish black as the reaction proceeded. Black sulfide heated in vacuo or in H at 700-800° became very much lighter and in some cases fragments of blue Si were obtained; while mixtures of amorphous Si and white SiS<sub>2</sub> sublimed at 1100-1250° gave black and yellow SiS. C. A. R.

Amorphous States of Silicon. Livio Cambi. Atti accad. Lincei, 20, I, 440-2; cf. preceding abstr.—The product obtained from the black sulfide differs in physical properties from the amorphous forms of Si thus far described; it has a lighter, reddish yellow color, gradually changes in properties on heating; after 1 hr. at 900° in vacuo, it assumes a brown color and the d. increases. The amorphous states of Si cannot be considered as distinct allotropic modifications; probably they are analogous to the amorphous states of C, a mixture of unstable forms tending to transform themselves into a stable form—the crystallin.

Chas. A. Rouiller.

Luminosity of Phosphorus. Lecture Experiment. L. MARINO AND C. PORLEZZA. Ist. chim. gen. r., Univ. Pisa. Atti accad. Lincei, 20, I, 442-6.—Red P is cautiously heated in a slow current of CO<sub>2</sub> until perfectly dry, then the flow of CO<sub>2</sub> increased and the heat raised until white P begins to distil over, when the vapors are passed into a balloon flask full of air. The P, coming in contact with the O, gives a green flame and the whole bottom of the flask becomes phosphorescent. If the P vapors and CO<sub>2</sub> are passed over into a bulb tube inclined at an angle of 25° at such a rate that a green flame constantly appears at the point where the exit tube enters the bulb tube, a yellow substance is deposited which is non-phosphorescent, does not evolve heat when treated with H<sub>2</sub>O, does not change in the air, reduces NH<sub>2</sub>-AgNO<sub>2</sub>, turns brown with conc. NH<sub>2</sub> but does not give PH<sub>3</sub>, takes fire at 80-9°, and contains about 84% P; i. e., it has approx. the comp. P<sub>4</sub>O.

Chas. A. Rouiller.

Existence of a New Type of Dioxides. Reaction between Selenious Acid and Manganese Dioxide. L. Marino and V. Squintani. Ist. chim. gen. r., Univ. Pisa. Atti accad. Lincei, 20, I, 447-52; cf. Z. anorg. Chem., 56, 234.—Manganese selenite, MnSe<sub>2</sub>O<sub>6</sub> orange-yellow cryst. powder, is obtained by cautiously boiling 25 g. moist MnO<sub>3</sub> with 70 g. SeO<sub>3</sub> in 50 cc. H<sub>2</sub>O, digesting the washed product 1 hr. with 200-250 cc. cold HNO<sub>2</sub> (7-8%), washing with acid and H<sub>2</sub>O and drying at 100°. It is practically insol. in H<sub>2</sub>O, HNO<sub>3</sub> and dil. H<sub>2</sub>SO<sub>4</sub>, sol. in HCl with evolution of Cl, liberates I from AcOH solns. of KI, forms MnO<sub>2</sub> with alk. hydroxide and carbonates, evolves CO<sub>2</sub> quantitatively from (CO<sub>2</sub>H<sub>1</sub>)<sub>2</sub>, oxidizes mercurous and cuprous salts, K<sub>4</sub>Fe(CN)<sub>6</sub> and As<sub>2</sub>O<sub>3</sub>. Me<sub>2</sub>SO<sub>4</sub> gave Me<sub>2</sub>O, a dense oily liquid with pungent color and a hygroscopic cryst. mass consisting chiefly of H<sub>2</sub>SeO<sub>6</sub>; the presence of an acid corresponding to dithionic acid is thus precluded. Heated at 400°, the salt is decomp. into SeO<sub>2</sub> and Mn<sub>2</sub>O<sub>4</sub>, MnSeO<sub>4</sub> being an intermediate product. The above facts serve to confirm the authors' views that PbO<sub>2</sub> and MnO<sub>2</sub> have different constitutions. C. A. R.

Tin Arsenides. N. PARRAVANO AND P. DE CESARIS. Ist. chim. r., Univ. Roma. Atti accad. Lincei, 20, I, 593-6.—The fusion diagram of the system Sn-As and micrographic study of the alloys indicates the existence of only 2 compds., Sn<sub>2</sub>As, and SnAs. Chas. A. Rouiller.

Solubility of Orthoboric Acid, its Molecular Weight and Transformation into the other Hydrates. R. NASINI AND F. AGENO. Gazz. chim. ital., 41, I, 131-6; cf. C. A., 4, 723.

C. A. ROUILLER.

Molecular Weight and Constitutional Formula of Sulfuric and Nitric Acids. GIUSEPPE ODDO AND GIOVANNI ANELLI. Pavia, ist. chim. gen. univ. Gazs. chim. ital., 41, I, 552-68; Chem. Zig., 35, 837-9, 846-7; cf. C. A., 5, 1744.—Mol. wt. detns. of Me<sub>3</sub>SO<sub>4</sub> in AcOH and C<sub>5</sub>H<sub>2</sub> having shown that it does not form double mols. the abnormal results obtained in H<sub>2</sub>SO<sub>4</sub> could not be explained on the basis of such double mols. of the ester but it must be the acid which exists in the form of (H<sub>2</sub>SO<sub>4</sub>)<sub>2</sub>. Detns. of the

vapor d. of the acid by the V. Meyer method in boiling anthracene (351°) gave the following results: Abs. acid, 101.9; acid with 4.482% H<sub>2</sub>O, 119.0, 100.3; with 7.447% H<sub>2</sub>O, 103.2; acid with 22.36% SO<sub>2</sub>, 97.3; with 19.61% SO<sub>2</sub>, 89.9. These results indicate the presence of (H<sub>2</sub>SO<sub>4</sub>)<sub>2</sub>, H<sub>2</sub>SO<sub>4</sub>, SO<sub>2</sub> and H<sub>2</sub>O mols. and, where the acid was not abs., of SO<sub>2</sub> and O<sub>3</sub>. Expts. having shown that f. p. detns. of the mol. wt. of HNO<sub>2</sub> in so-called "associating" solvents (PhNO<sub>2</sub>, C<sub>2</sub>H<sub>4</sub>Br<sub>2</sub>), as well as in AcOH and CH<sub>2</sub>ClCO<sub>2</sub>H, gave values only slightly higher than those required by the simple formula, the expts. were repeated with H<sub>2</sub>SO<sub>4</sub> in the same solvents (except C<sub>2</sub>H<sub>4</sub>Br<sub>2</sub>) and in HCO<sub>2</sub>H, correction being made for the small amts. of H<sub>2</sub>O present in the solvents, and in all cases values corresponding to the double formula were obtained. A systematic study of compds. of the following type (M = metallic or org. bases) were also made: MH<sub>2</sub>SO<sub>2</sub>, MM'M"HS<sub>2</sub>O<sub>2</sub>, isomeric salts MM'M"M"'S<sub>2</sub>O<sub>2</sub>, mixed salts, etc. The results of this investigation will be published later. The authors conclude that the only form of H<sub>2</sub>SO<sub>4</sub> thus far isolated is that corresponding to the fibrous anhydride S<sub>2</sub>O<sub>4</sub> (Bull. soc. chim., [3] 25, 897) and has the formula (I). A second possible formula (II) seems

excluded by the fact that such a compound ought to have a geometric isomer (III) which should be detected by differences in the f. p. of the abs. acid or by the transformations to which such compds. in org. chemistry give rise.

C. A. ROUILLER.

The Preparation and Determination of Nitric Oxide and its Behavior with Water. L. Moser. Z. anal. Chem., 50, 401-33.—The author has made an exhaustive study of the methods that have been proposed for the prep. and analysis of NO and the changes it undergoes in contact with water. A closed form of absorption app. is described in which intimate contact of gas and absorbing liquid is attained with complete exclusion of air. The following conclusions are reported: 1. The best methods for the prep. of pure NO are the Lunge-Emich (Monatsh., 13, 73), based on the reduction of HNO, (in H2SO4) by Hg and the Winkler (Ber., 34, 1408), based on the reaction between HI and HNO. 2. NO cannot be kept over water without undergoing changes which are effected by the dissolved O and by the H ions present. HNO, is reduced to H<sub>2</sub>N<sub>2</sub>O<sub>2</sub> which decomposes in part to N<sub>2</sub>O and in part to NH<sub>2</sub>, which forms NH<sub>4</sub>NO<sub>2</sub> and breaks down into N, which is always present in a large amt. as a decomp. product when kept over water for a long time. 3. Moist or dry NO can be kept indefinitly at room temp. without decomp. over Hg. 4. The absorption of NO by FeSO, soln. is complete; the results are erroneous if NO<sub>2</sub> is present simultaneously. 5. The detn. of NO by alk. Na, SO, soln. has the disadvantage that the absorption is complete only after long shaking on account of the small velocity of the reaction between the NO and SO, ions. 6. The volumetric detn. of NO with KMnO, in the usual way, using an inert gas, easily gives low results on account of the low reaction-velocity between the NO and MnO4 ions—very small amts. of NO may not be detd. By using the specially devized absorptiometer accurate results are quickly obtained and the error due to inert gas is avoided. 7. Using this app. accurate results are obtained by oxidizing NO with H<sub>2</sub>O<sub>2</sub> and detg. alkaline alkalimetrically the HNO<sub>2</sub>. Acid gases or vapors must be removed. 8. The method of combustion of the NO mixed with H gives erroneous results due to side-reactions in which NH, compds. are formed and to the perviousness of red hot Pt tubes. It is shown that thru the compensation of these errors F. W. SMITHER. correct results may be obtained.

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Bismuth Carbonate. L. Vanino. Pharm. Zentralhalle, 52, 761-2.—The comp. of the basic carbonate o Bi was assumed to be (BiO)<sub>2</sub>CO<sub>2</sub> with a variable proportion of water. By pouring a conc. soln. of Bi(NO<sub>2</sub>)<sub>2</sub> into a strong soln. of Na<sub>2</sub>CO<sub>2</sub>, washing and drying the ppt., the product has the comp. 5Bi<sub>2</sub>O<sub>3</sub>.Bi<sub>2</sub>(CO<sub>2</sub>)<sub>3</sub>. BiCl<sub>3</sub> soln. gave halogen-containing ppt. A soln. of Bi(NO)<sub>3</sub> in mannitol, 48.2: 18.2, is clear but decomposes if no H<sub>2</sub>O is used. Mixed with a satd. soln. of K<sub>2</sub>CO<sub>2</sub> at o°, CO<sub>2</sub> was evolved, and a fine ppt. sepd. When washed and dried, the ppt. was found to contain 81.12% of Bi (theory 81.89%). With (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> in place of K<sub>2</sub>CO<sub>3</sub> the product was (BiO)<sub>2</sub>CO<sub>3</sub>. A com. sample (Kahlbaum), free from acids and H<sub>2</sub>O, gave 81.26 and 81.54% Bl, therefore (BiO)<sub>2</sub>CO<sub>3</sub>.

Action of Electrical Discharge on Ammonia (BESSON). 2.

Double Salts of Antimony Pentachloride and Alkaloid Hydrochlorides (THOM-SEN). 10.

Extraction of Sulfur from Minerals (ROCASOLANO). 18.

Fusion of Silicates (DITTLER). 19.

Second Spectrum of Hydrogen (PORLEZZA, NORZI). 2.

Solutions of Sodium Aluminate (SLADE). 29

# 7. ANALYTICAL CHEMISTRY.

E. G. R. ARDAGH.

The General Essentials in Teaching Qualitative Analysis. H. C. COOPER. Syracuse Univ. Science, 34, 263-5.

H. M. LANCASTER.

A New Method of Chemical Analysis. SIR J. J. THOMSON. Chem. News, 103, 265-8; Engineering, 91, 489-90; Met. Chem. Eng., 9, 298-9.—A discourse delivered before the Royal Institution, April 7, 1911.

W. H. Ross.

Some Principles of Physical Chemistry of Importance for Quantitative Chemical Analysis. Enrique Hauser. Lab. Escuela Minas. Anales soc. españ fis. quim., 8, 258-61.—The author, in emphasizing the importance for quant. analysis of a study of chem. equil., as developed in modern physical chemistry, discusses some cases of the establishing of the equil. between a ppt. and salts in soln. by means of double decomp. and formation of double salts. As showing how loss of ppt. by formation of double salts may be avoided by diln. sufficient to secure a high degree of dissociation of the salts present the author instances this method (Anales soc. españ. fis. quim., 1904, p. 301) for sepn. of As and Cu; if not more than 50 mg. of Cu be present in 100 cc., complete pptn. of the Cu may be obtained by making the soln. (best kept at 50°) slightly ammoniacal and passing a current of H.S. For avoiding equil. disturbances in the pptn. of Mg salts by baryta water in the presence of alk. chlorides the following procedure is recommended as more readily carried out than the methods commonly used. The first pptn. will be practically complete if made in a sufficiently dil. soln. (about 2: 1000). Next ppt. the Ba, expel NH4 salts, ppt. most of the NaCl present by conc. HCl (Treadwell, Quant. Analysis, 1907, p. 42) and ppt. the remaining Mg in the dild. filtrate as at first. If alk. salts are present in very great amt., they should be pptd. before making the first Mg pptn. This procedure does not interfere with detn. of Na or K.

Advances in Microchemistry Since H. Behrens. I. F. EMICH. Chem. Ztg., 35, 637-9.—A résumé, with references, of the later work in the use of the microscope in chemistry. Electrochemical detns. of small amts. of material are discussed. Spectroscopic and polarimetric detns. of liquids enclosed in capillary tubes, sp. gr. of liquids and solids in small pycnometers, indices of refraction of liquids and crystals are com-

mented upon. *Ibid.*, 663-5.—(A) Qual. inorganic analysis: A discussion of methods of microanalysis. Many of the fusion and pptn. reactions of macrochemistry are done microchemically. *Organic analysis:* This includes the work of Ostwald on oils and gelatin, Raciborski on proteins, amino acids, phenols, pyrrole, indole and skatole, and of Bolland on the organic bases. (B) *Quantitative:* Emich and Donau have obtained results with an error of never more than 5%.

J. A. WILKINSON.

Detection of Chlorides in Bromides and Iodides. ANDRE TESTA. Turin. Ind. chim., 11, 123-4.—The soln., slightly acid with HNO<sub>2</sub>, is pptd. with AgNO<sub>2</sub>, the ppt. washed free of sol. Ag salts by decantation, then boiled about 1 min. with 10 cc. 0.5 N NH<sub>4</sub> oxalate, the boiling liquid filtered and a few drops of KBr added. If any chlorides were present in the original soln., a yellowish white turbidity, due to AgBr, persisting even on b., is formed; as little as 0.0002 part HCl can thus be detected. HCNO, HCNS or H<sub>2</sub>Fe(CN)<sub>4</sub> must, however, not be present. C. A. ROUILLER.

A Method of Separating the Metals of the Ammonium Carbonate Group. Calcium, Barium and Strontium. ERICH BIRNBRÄUER. Chem. Ztg., 35, 755.—The carbonates, obtained in the ordinary way, are washed with hot  $H_2O$ , dissolved in dil.  $HNO_2$ , evapd. on the  $H_2O$  bath, taking care that no nitrate is decomposed, the nitrate treated with a warm 1: I Et<sub>2</sub>O-EtOH mixt. and the Ca(NO<sub>3</sub>)<sub>3</sub> soln. filtered off. If the first portion shows the presence of Ca, the treatment is repeated, and the Ba and Sr remaining washed 3 times with the same mixt. Ca is detd. in the filtrate with  $(NH_4)_2C_2O_4$ . The Ba and Sr nitrates are dissolved in a little  $H_2O$ , NaOAc and a little AcOH added, the Ba pptd. as BaCrO<sub>4</sub>, and the Sr pptd. from the filtrate with an equal vol. 1: 2  $(NH_4)_3$ -SO<sub>4</sub> soln. The SrSO<sub>4</sub> is sol. (1: 17,000) in the 20%  $(NH_4)_2SO_4$  soln. resulting. No results are given.

The Extraction of Bromine and Iodine Simultaneously Liberated in Aqueous Medium by means of Chloroform and Carbon Disulfide. A. LABAT. Laboratoire de chim. biologique de la Faculté de Méd. et de Pharm. de Bordeaux. Bull. soc. chim., 9, 503-6.

—A study has been made of the methods that have been published by previous investigators and the following résumé is given by the author: In a mixture of alk. bromide and iodide in soln., if it is desired to detect the halogens by liberatingwit han oxidizing agent and dissolving in CHCl<sub>2</sub> or CS<sub>2</sub>, Cl should not be used as the I may escape detection; NaNO<sub>2</sub> is of use for I only and is rather detrimental for Br (a nitrosyl of Br is formed which is decomposed readily by water into HNO<sub>2</sub> and HBr). A reagent capable of liberating the 2 elements at the same time should be in the proportion of one of the elements and then sometimes the 2 are not detected.

F. W. SMITHER.

Determination of Arsenic and Iron Salts in Mineral Waters. F. AGENO AND N. GUICCIARDINI. Pisa ist. chim. gen. univ. Gazz. chim. ital., 41, 1, 473-8.—The total ferrous salts, arsenites and org. matter are first detd. by oxidation in acid soln. with KMnO4, then the total As and Fe, then the ferrous salts by the Fresenius BaCO4 method, the H<sub>2</sub>SO4 being first removed by pptn. in slightly acid (HCl) soln. in an atm. of CO4, thus preventing oxidation of the ferrous salts, and finally the arsenites by titration with I, the Fe being first pptd. with alk. hydroxides and carbonates and the filtrate treated with CO4 to convert the alk. into the bicarbonates. The best method for detg. the total As is that of Andrews and Faw (Z. anorg. Chem., 62, 123). The results of analysis of a soln. containing 0.2139 g. to al As, 0.1068 As as arsenites, 0.1071 as arsenates, 0.3194 total Fe, 0.1518 ferrous Fe, 0.1676 ferric Fe and 0.1534 org. matter (oxalic acid) were 0.2142, 0.1050, 0.1092, 0.3200, 0.1534, 0.1666, 0.1490, resp.

CHAS. A. ROUILLER.

A Reaction for Uranium Salts. J. A. SIEMSSEN. Chem. Ztg., 35, 742; cf. C. A., 5, 1886.—The ppt. obtained when 10% ethylenediamine soln. is added to a soln. of a

U salt settles rapidly, is easily filtered, is insol. in Me<sub>2</sub>CO, PhH, Et<sub>2</sub>O, EtOH, C<sub>2</sub>H<sub>11</sub>OH, CHCl<sub>2</sub>, CS<sub>2</sub>, MeOH, PhMe, PhEt and petroleum ether. The ppt. is washed with cold H<sub>2</sub>O, ignited moist in Pt, weighed as U<sub>2</sub>O<sub>4</sub>. The soln. should be fairly concentrated, otherwise the ppt. is gelatinous. Other metals give ppts. with the same reagents. No results are given.

George W. Morey.

Notes on the Analysis of Tungsten Ores. Hugh F. Watts. Boulder, Colo. Met. Chem. Eng., 9, 414-5.—When W ores have been decomp. by fusion or digestion with alkalis, it is essential that the WO<sub>3</sub> be pptd. with HgNO<sub>3</sub>; evapn. with HCl gives incorrect results. The author gives both the Na<sub>2</sub>CO<sub>3</sub> fusion and the aqua regia treatment for opening up the minerals, using the latter method for ores of the Boulder district. In the first case he ppts. with HgNO<sub>3</sub>, in the second dissolves the WO<sub>3</sub> from the SiO<sub>2</sub>, etc., with NH<sub>4</sub>OH, and obtains it in we ghable form by evapn., ignition, and treatment with HF.

George W. Morey.

Analysis of Manganese Bronze. J. R. Huber. Met. Chem. Eng., 9, 403.—A full description of a useful method. Not abstractable.

F. C. Phullips.

Comparison of Methods for the Determination of Nickel in Steel. J. J. BOYLE. American Bridge Company, Ambridge, Pa. Chem. Eng., 14, 288-9.—The author compares the dimethylglyoxime method (weighing the ppt. dried at 110-20°) with the cyanide titration method and concludes that the former is the more accurate. Details of his procedure are given in full.

F. C. PHILLIPS.

Examination of Tin in an Ore. R. J. Morgan. Chem. Eng., 14, 289-91.—The ore is fused with NaOH in an iron crucible, the fusion after softening in water is decomposed by HCl. The SnCl, and FeCl, are reduced by Fe (horse shoe nails) and the soln. filtered. Powdered Sb is added, the soln. boiled and then cooled while a stream of CO, is being passed and finally SnCl, is detd. by standard I soln. The author prefers this method of titration to that by standard FeCl, soln.

F. C. PHILLIPS.

Simultaneous Volumetric Determination of Iron and Vanadium. E. MULLER AND O. DIEFENTHALER. Z. anorg. Chem., 71, 243-9.—In the effort to improve Campagne's method for V, the authors found that evapn. of V solns. with HCl and EtOH in an open dish, followed by furning with HaSO4, diln. and titration with KMnO4, lead to low results, due to incomplete reduction. The same operation carried out in a flask yielded high results, due to incomplete expulsion of organic compds. The latter effect could be ovecome by heating the soln. on a water bath for 15 min., stirring the moist residue at intervals. The residue could then be taken up in H,SO4, without heating, and titrated as in the Zimmerman-Reinhardt method for Fe (addn. MnSO, and H<sub>2</sub>PO<sub>4</sub>). The authors give the following method for the analysis of ferrovanadium: Dissolve 1 g. in HNO, in a beaker, evap. with HCl to expel HNO, add 20 cc. conc. HCl and 50 cc. EtOH, heat gently, and finally evap. an a water bath to 5 cc. Dilute to a definit vol.; det. Fe iodometrically in one aliquot, and V in another portion by titration in the cold with 0.1 N KMnO4 after addition of MnSO4 and H2PO4. Instead of using conc. HCl and EtOH, a satd. soln. of gaseous HCl in 96% EtOH may be used. Results for V are accurate, those for Fe about 0.5% high. WILLIAM BLUM.

The Separation of Iron and Vanadium by the Ether Method. E. Deiss and H. Leysaht. Chem. Zig., 35, 869–71.—The Rothe method, as executed, gives low results. In the  $V_2O_4$  state some of the V is held in soln. by the Et<sub>2</sub>O giving a yellow soln. By reducing to  $V_2O_4$  with SO<sub>2</sub> no V was dissolved in Et<sub>2</sub>O until after several days. The time required in analysis for a sepn. is so brief, however, that any error due to soln. of  $V_2O_4$  is negligible. It is shown that the reduction to  $V_2O_4$  by repeated evapn. with HCl is complete with only very small amts. of V. Ordinary Et<sub>2</sub>O, on account of the peroxide it contains, changes the pure blue color of HCl soln. of  $V_3O_4$  to

blue-green or green due to formation of pervanadic acid which is insol. in Et<sub>2</sub>O. As SO<sub>2</sub> would also reduce Fe'' and Fe'' is insol. in Et<sub>2</sub>O, it is recommended to remove most of V in the usual way and then to shake out the Et<sub>2</sub>O soln. of FeCl<sub>2</sub>—containing remainder of V with HCl (d. 1.10), satd. with Et<sub>2</sub>O and H<sub>2</sub>O<sub>2</sub>. For the titration of the main amt. a KMnO<sub>4</sub> soln. (1 cc. = 0.006292 g. V) was used, for the small amts. a soln. of which 1 cc. = 0.001014 g. V.

F. W. SMITHER.

The Use of "Hyperol" in the Laboratory. J. MILBAUER. Chem. Ztg., 35, 871.-"Hyperol" is a solid "H<sub>2</sub>O<sub>2</sub> prepn." that may be used to replace H<sub>2</sub>O<sub>2</sub> solns., is quite stable and easily handled. It is described as a compd. of H<sub>2</sub>O<sub>2</sub> with urea, containing a little citric acid to render stable and may be had either in a fire cryst. powder or in tablets. Analysis gave: urea 65.70%, H<sub>2</sub>O<sub>2</sub> 34.00%, citric acid 0.08%. Theory for CO(NH<sub>2</sub>)<sub>2</sub>.H<sub>2</sub>O<sub>2</sub> requires 35.9% H<sub>2</sub>O<sub>2</sub> and 64.1% urea. It dissolves in water (up to about 60%) and in EtOH, Et<sub>2</sub>O causes evolution of H<sub>2</sub>O<sub>2</sub>; on ignition it leaves only a trace of ash. It is free from H<sub>\*</sub>SO<sub>4</sub> and at 60° begins to decomp., O escaping and water and urea remaining. In aq. solns. of chromates it produces (after adding H<sub>2</sub>SO<sub>4</sub>) the blue perchromic acid; in alk. solns. it oxidizes Cr<sub>2</sub>O<sub>2</sub> to CrO<sub>2</sub>. Sulfides are oxidized quantitatively to sulfates, Fe" to Fe". It may be used without hesitation for H<sub>2</sub>O<sub>2</sub> in the quant. sepn. of metallic salts by Jannasch and for the estn. of volatil S in fuels by Hempel's method, in which the NO is destroyed by the urea present. Solns. of Au salts are colored violet. It may be used for detg. insol. matter in red lead, for distinguishing between fresh and boiled milk, for detn. of HCOH, for sepn. of Ti and Fe as well as Fe and Zn from U. The fact that it gives up H<sub>2</sub>O<sub>2</sub> in Et<sub>2</sub>O might find a practical significance in "Katatypie." The material is made by Gedeon Richter, Budapest. F. W. SMITHER.

A Practical Method for the Reduction of Potassium Chloroplatinate for the Determination of Potassium. A. Fiechter. Z. anal. Chem., 50, 629-32.—The aq. soln. of the K<sub>2</sub>PtCl<sub>2</sub> ppt. is reduced with Mg filings or ribbon on presence of a little HCl, the excess of Mg afterwards dissolved in HCl and the Pt filtered, ignited and weighed on Gooch crucible.

F. W. Smither.

Titration of Potassium Cyanide in the Presence of Potassium Ferrocyanide. W. D. TREADWELL. Charlottenburg. Z. anorg. Chem., 71, 219-25.—Liebig's method for the direct titration of KCN with AgNO, is not applicable in the presence of K, Fe-(CN)<sub>a</sub>. If, however, o. 1 g. KI is first added to the soln., which should be about o. 1 N and slighlty alk., accurate results are obtained. The addition of NH<sub>4</sub>OH, as recommended by Deniges and Sharwood, is not necessary. The disturbing influence of Na<sub>2</sub>S<sub>2</sub>O<sub>2</sub> may be overcome by increasing the amt. of KI to 1.0 g. The author found a satisfactory explanation of the above facts in a study of the (Ag+) in the various solns. This was detd. from measurements of the e. m. f. of a conc. cell consisting of o. 1 N AgNO<sub>2</sub>, and the soln. to be tested. In the titration of pure KCN solns. with AgNO<sub>2</sub>, it was found that upon the addition of the drop before the appearance of a turbidity, there was a sudden decrease in the e.m. f. from 0.724 to 0.523 v, due to an increase in (Ag+). Similarly the influence of excess NH, of Na<sub>2</sub>S<sub>2</sub>O<sub>2</sub>, and K<sub>4</sub>Fe(CN)<sub>6</sub> was found to be due to the formation of complex ions, and the lowering of (Ag+) below the conc. for pptn. of AgCN. Addition of KI caused in each case a turbidity due to the lesser WILLIAM BLUM. solubility of AgI.

Action of Hydrazine Sulfate upon Nitrites, and a New Method for Determining Nitrogen in Nitrites. B. B. Dey and H. K. Sen. Z. anorg. Chem., 71, 236-42.—By measurement and analysis of the gas evolved in a nitrometer, the authors showed that in the action of  $N_2H_4$  (as sulfate) upon nitrites the following reactions take place simultaneously and to the same extent:  $N_2H_4 + 2HNO_2 = N_2 + N_2O + 3H_2O$  and  $N_2H_4 + HNO_2 = NH_2 + N_2O + H_2O$ , i. e.,  $NH_2$  remains in the form of sulfate and

the gas mixt. contains  $N_2$  and  $N_2O$  in the ratio of 1:2. (In the  $N_2O$  detns. it was necessary to correct for the solubility of the  $N_2O$  in the vol. of water present.) This ratio was found to hold for  $Ba(NO_2)_2$ ,  $KNO_2$ ,  $AgNO_2$ ,  $N(CH_2)_4NO_2$  and  $C_2H_3CH_2NH_2NO_2$ , but not for  $C_3H_{11}NO_2$  (which is not ionized). For the detn. of nitrite N this method is as accurate as the urea method, the procedure being to treat the nitrite soln. with solid hydrazine sulfate, collect the evolved gas, dissolve out all the  $N_2O$  with  $H_2O$ , and measure the residual  $N_2$ , which is equal to  $^2/_2$  of that present in the nitrite. W. Blum.

A Simple Method for Obtaining the Spectroscopic Test for Hemochromogen in Blood Stains. D. Mirro. Ist. Med. Leg., Univ. Catania. Arch. farm. sper., 12, 114-8.——In place of the usual method of converting the hemoglobin to hematin and then reducing to hemochromogen before applying the spectroscopic test, the author recommends the use of a 25% alc. soln. of phenylhydrazine by which this change is brought about in one step.

A. W. Dox.

Separation and Determination of Ammonia and Pyridine. M. Delépine and R. Sornet. Bull. soc. chim., 9, 706–10.—In the analysis of complex compounds of Ir, the following method was finally adopted: NH<sub>2</sub> and C<sub>2</sub>H<sub>2</sub>N were liberated from the complex by Na<sub>2</sub>S, and distilled into a slight excess of HCl. To this soln. was added successively 50–100 cc. HgCl<sub>2</sub> (30 g. per liter), 30 cc. Na<sub>2</sub>CO<sub>3</sub> (200 g. per liter), and 5–10 cc. NaOH (400 g. per l.). The ppt. formed contained all the NH<sub>2</sub>. The soln. was agitated, diluted to a known vol., allowed to settle, filtered and an aliquot portion of the filtrate used for the detn. of C<sub>5</sub>H<sub>3</sub>N. It was evaporated with excess of either AuCl<sub>2</sub> or PtCl<sub>4</sub>, taken up in EtOH and Et<sub>2</sub>O, filtered, washed, and ignited to Au or Pt, from which was calculated the C<sub>5</sub>H<sub>3</sub>N. The ppt. containing Hg and NH<sub>2</sub> was washed with water containing 10 g. Na<sub>2</sub>CO<sub>3</sub> + 10 g. NaOH per l., and was finally distilled with 50 cc. Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (200 g. per l.). The NH<sub>3</sub> was absorbed in HCl, evapd. to dryness, dried at 105° and weighed as NH<sub>4</sub>Cl. Several analyses of known mixtures gave satisfactory results.

WILLIAM BLUM.

An Analytical Application of some Kanthogenates. Jaime Ferrer and Angel Del Campo. Anales soc. españ. fis. quim., 9, 173-4.—The authors in a preliminary note state that an exam. of a no. of different alkyl xanthogenates indicates several that may be of value for differentiating Ni and Co. K propylxanthogenate, for instance, is adapted to a color reaction; it gives a yellow ppt. with Ni salts and green with those of Co. A solubility difference may be obtained by use of K methylxanthogenate. The Ni ppt. is very sol., while the Co ppt. is only slightly sol. in NH<sub>3</sub> water; if NH<sub>3</sub> water of suitable diln. be used, the liquid remains clear if only Ni is present and becomes turbid on presence of even a minute amt. of Co. The authors expect to report further on the efficacy of these tests.

H. S. Paine.

Agitators for Determinations of Ag, P and Mg (BAHNEY). 1.

Analysis of Artificial Pearls and Rubies (CERERO, BAYO). 8.

Analysis of Oils by Miscibility Curves (LOUISE). 27.

Colorimetric Determination of Dyes (EIJDMAN). 25.

Colorimetric Estimation of Sugar (AUTENRIETH, MULLER). 11.

Determination of Ketones in Essential Oils (NELSON). 17.

Determination of Malic Acid (DUNBAR, BACON, YODER). 12.

Determination of Pb and Zn in Rubber Goods (KUHL). 26.

Determination of Reducing Sugars (MULLER). 28.

Preparation and Determination of Nitric Oxide (MOSER). 6.

Second Spectrum of Hydrogen (PORLEZZA, NORZI). 2.

The Turmeric Test for Boric Acid (PRICE, INGERSOLL). 12.

The Wortman Formula (FALLADA). 28.

## 8. MINERALOGICAL AND GEOLOGICAL CHEMISTRY.

### ROGER C. WELLS.

Double Refraction of Sodium Chlorate. Georges Meslin. Compt. rend., 152, 1666-8.—The author studies the circular polarization of light by liquids and substances crystg. in the cubic system by means of a Fresnal triprism built up of an isosceles prism of dextrorotatory NaClO<sub>2</sub> crystals and selected crown-glass prisms. The NaClO<sub>3</sub> crystals were prepd. by fusing a mass of the substance and sepg. the right and left crystals mechanically. Using the Hg arc as a source of light, the author was able to sep. yellow, green, and violet rays each into two circularly polarized rays.

GEORGE W. MOREY.

Radioactive Equilibrium in Vesuvius Cotunnite. PAOLO ROSSI. Att. accad. Lincei, 19, II, 578-83; Chem. Zentr., 1911, I, 751.—In a Vesuvius cotunnite containing Ra D, E and F found in 1907, besides Ra D, there were in Dec., 1910, Ra E and F not in equil. with the Ra D. This is in agreement with Piutti's observation (C. A., 4, 3055) that there is no He in cotunnite.

Results of the Chemical Investigation of an Uranium Mineral from Borneo. G. TSCHERNIK. Petersburg Chem. Lab. Akad. Wissenschaften. Bull. Acad. St. Petersburg, 1909, 1203-12; thru Chem. Zentr., 1910, I, 1287.—The mineral proved to be a bröggerite. Crystals, well formed and of dark color; hardness, 5-6; d<sub>16</sub>, 9.057; radioactive; easily sol. in aqua regia. The av. comp. was: UO<sub>3</sub>, 40.91; UO<sub>2</sub>, 41.19; PbO, 8.50; ThO<sub>2</sub>, 5.01; Y<sub>2</sub>O<sub>3</sub>, 1.56; CaO, 0.18; SiO<sub>2</sub>, 0.76; FeO, 0.90; H<sub>2</sub>O, 0.52% and traces of Bi<sub>2</sub>O<sub>3</sub>, MgO and CuO. The ratio of the rare earths as they occur in bröggerite was detd.: Ce<sub>2</sub>O<sub>3</sub>: La<sub>2</sub>O<sub>3</sub>: Nd<sub>2</sub>O<sub>3</sub>: Pr<sub>2</sub>O<sub>3</sub> = 8:5:1:2. For comparison a bröggerite from Moss, Norway, was analyzed with results to which the above agree.

E. J. CRANE.

Technic of the Analysis of Artificial Pearls and Rubies. RAFAEL CERERO AND ENRIQUE BAYO. Lab. escuela minas. Anales soc. espan. fis. quim., 9, 178-81.—The following comp. was found for artificial pearls: Moisture (at 100°) 0.40%, CaCO2 89.10, Fe<sub>2</sub>O<sub>2</sub> 0.43, CaSO<sub>4</sub> 2.72, organic matter and combined H<sub>2</sub>O 7.35%. For artificial rubies: Al<sub>2</sub>O<sub>2</sub> 94.00%, Cr<sub>2</sub>O<sub>2</sub> 1.70, Fe<sub>2</sub>O<sub>2</sub> 1.15, SiO<sub>2</sub> 2.80, MgO traces. A mixt. of borax and alk. carbonates was found best for fusing the rubies when a Pt crucible is used. It is observed that Ca phosphate, usually present in natural pearls, is lacking in the artificial ones examined. On pulverizing the pearls, extracting with alc. and ether, and evapg, there was obtained organic material of a resinous appearance readily oxidized by KMnO4. The % of organic matter was detd. by deducting H2O and CO<sub>2</sub> (detd. directly) from the loss of wt. on ignition. In both sp. gr. and hardness of the outer crust both pearls and rubies simulated the natural stones. The rubies fluoresced when exposed to cathode rays and a change of color from red to bottlegreen resulted, on raising the temp. sufficiently. Microscopical was found better than chem. exam. for differentiating natural and artificial pearls. The irregular concentric layers in natural pearls, due to successive secretions by the mollusk, were lacking in the artificial ones. From bubbles (distinct from the inclusions found in the natural stones) and concentric zones observed in the rubies the authors conclude that the stones examined were made by the process perfected by Verneuil. H. S. PAINE.

Existence of Alunite in the Liperite of Torniella in the Province of Grosseto. Ugo Paniche. Atti accad. Lincei, 19, II, 656-64; Chem. Zentr., 1911, I, 837.—Contrary to the views thus far held, the substance found in certain localities described is an alum, not a kaolin.

Chas A. Rouiller.

Deposits of Manganese in Lower California. H. V. WALLACE. Mining World, 35, 103-4.—The two deposits described, lying near Mulege, were probably deposited from solns. but the Guadalupe series is in layers, while the Gabilan deposits occur as fillings in a brecciated pyroxene. The author ascribes the latter development to metasomatic processes.

R. C. WELLS.

The Association of Lead with Uranium in Rock Minerals, and its Application to the Measurement of Geological Time. ARTHUR HOLMES. Imperial Coll. Sci., London. Proc. Roy. Soc. London, (A) 85, 248-56; cf. Boltwood, C. A., 1, 817.—The author made detns. of U (using Strutt's method in which Ra emanation is directly measured) and of Pb by grav. and colorimetric methods (Harcount, C. A., 4, 2617)) is a number of selected fresh stable primary rock-minerals; he found that the ratio Pb/U is nearly const. for minerals of the same age, the slight variability being what one theoret cally would anticipate, and that it increases for minerals of increasing geological age, as shown by the following table, in which the first figure represents the ratio of Pb/U, the second the age in millions of years, calc. from the known rate of production of He from U and the equation U -> 8He + Pb: Carboniferous, 0.041, 340; Devonian, 0.045, 370; pre-Carboniferous, 0.050, 410; Silurian or Ordovician, 0.053, 430; pre-Cambrian, (a) Sweden, 0.125-0.155, 1025-1270; (b) U. S. A., 0.160-0.175, 1310-1435; (c) Ceylon, 0.20, 1640. Wherever the geological evidence is clear, it is in agreement with that derived from the ratio Pb/U as an index of age; where it is obscure, as in the pre-Cambrian rocks, the evidence does not, at least, contradict the age derived in this way. This mode of calculation is justifiable only when the following conditions hold: (a) that no appreciable amt. of Pb was present when the mineral was formed; (b) that no Pb was originated by any other radioactive process; (c) that no Pb or U has subsequently been added or removed by external agencies. Owing to uncertainty in this regard, some of the detns. were not made use of in the calculations embodied in the above table. J. JOHNSTON.

Genesis of Bolivian Tin Deposits. M. Armas. Eng. Mining J., 92, 311-4.—An introductory review of geographical and geological features of the Andes mountains, having a bearing upon the subject, is presented. Veins containing Sn deposits usually occur in slate or quartz formations at great altitudes, where diminished pressures existed. The theories of Van Hise and Sandberg as to the origin of metallic deposits do not explain the deposition of Sn. The author advances the theory that under great temp. and pressure at lower levels, gaseous compds. of Sn were formed with certain metalloids, such as P or Si. On ascending into a zone of diminished temp. combination with F with the formation of a double salt is possible. Still higher H<sub>1</sub>O vapor causes the pptn. of SnO<sub>2</sub>. Sn formations in Bolivia are invariably connected with acid rocks of igneous origin mostly of the granitic type. The mineralogical features of the chief Sn deposits in Bolivia are reviewed.

# o. METALLURGY.

WILLIAM BRADY, WILLIAM T. HALL.

Power Requisit of Rolling Mills. J. Puppe. Stahl u. Eisen, 31, 372-8.

J. J. M.

Slime Settler or Dewaterer. R. E. HUNTLEY. Mines and Minerals, 31, 339.

J. J. M.

Welding up of Blow-holes and Cavities. J. E. STEAD. Iron and Steel Inst., May, 1911; Engineering, 91, 635-7; J. Soc. Chem. Ind., 30, 625.—Defines welding as the crystn. into union of 2 solid metallic surfaces under suitable conditions. If true welding has taken place, the microscope will show the joining crystals to be common to both pieces. Accounts of several series of expts. of welding together solid pieces of steel, and pieces with artificial blow holes (holes boared and partly plugged), pipes and blisters are given. Steels of varying C content were used. If in ide blow-holes in steel ingots have clean walls and contain no foreign matter, they will weld up completely during forging or rolling at temps. of 1000° or over. S. doubts whether pipe cavities can be so welded, as the walls generally contain high percentages of the impurities of the steel and the upper ends are usually open to gases which oxidize.

L. W. SPRING.

Titanium and its Benefit in Iron. CHARLES V. SLOCUM. Chem. Eng., 14, 327-9.
—Small quantities of Ti produce practically as good effects as large quantities. Addition of 0.02% of metallic Ti is frequently sufficient for cleansing the Fe by removal of the oxides and nitrides and gives it increased fluidity and less shrinkage which results in better and sharper castings, and the absence of blow-holes and the close grain of the metal give greater strengths. Use of Ti increases cost of the steel \$0.25-\$3.00 per ton.

L. W. Spring.

Pure Irons from the Open-hearth Furnace. ALLERTON S. CUSHMAN. Chem. Eng., 14, 330-2.—The electrolytic theory of metal corrosion has caused the production of the so-called "ingot iron," 99.80% or over Fe. It is made in the open-hearth furnace by completely burning out the Mn and C, then by heroic treatment so completely deoxidizing the bath that metal of not over 0.03% O and 0.01% N results. The slagless metal is not steel in the usual sense of the word nor is it iron, as the slag-enclosing product of the charcoal knobling fire at the puddling furnace is called, but it is more entitled to that name than is the wrought iron, which contains 3% or so of slag. Ingot iron contains 0.02-0.08% Cu as is shown by analyses given. Expts. on irons containing up to 1.5% Cu show that no difficulty is encountered in rolling and finishing such alloys. Cu in the metal retards its soln. in dil. mineral acids. "Ingot iron" is necessarily somewhat more expensive than steel, owing to the higher temps., more prolonged heating and greater care needed for its manuf. It has so far been successfully used for billets, slabs, plates, sheets, merchant iron, wire nails, rivets, pipe, boiler tubes and forging and should be well adapted for drawing and stamping pur-L. W. SPRING. poses.

Blast Furnace Slag. 20.

Corrosion of Steel. 20.

Extraction of Gas from Copper (GUICHARD). 6.

Mitigation of Electrolysis (LAMPHER, SMITH). 14.

Magnetic Constants of Feebly Magnetic Alloys (GNESOTTO, BINGHINOTTO). 2.

Tin Arsenides (PARRAVANO, DECESARIS). 6.

Ger., 236,006, Sept. 24, 1908. J. CHURCHWARD, Mount Vernon, U. S. A. In the heating and hardening of steel alloys, subjecting the alloy to a series of gradually decreasing heatings and rapid coolings. Further details are specified.

Ger., 236,007, Mar. 19, 1908. A. W. MACHLET, Elizabeth, U. S. A. Production

of a gas mixture, consisting of C-containing and of indifferent gases, serving for the cementation of steel or iron, by leading the gas or gases of the one kind thru liquids which yield the gases of the other kind. E. g., the indifferent gases are led thru C-contg. liquids, as HN, thru oil, naphtha, or the like, or C-contg. gases, as illuminating gas, are led thru containers filled with liquids which give off indifferent gas, such as ammonia H<sub>2</sub>O.

### 10. ORGANIC CHEMISTRY.

J. BISHOP TINGLE.

New Organic Compounds of Nitrogen. M. O. Foster. Engineering, 91, 624-5.
J. J. M.

Chemical Investigation of the Oil of Chenopodium. E. K. Nelson. J. Am. Chem. Soc., 33, 1404–12; U. S. Dept. Agr., Bur. Chem. Circ. 73.—Chenopodium oil  $(d_{26}^{26} 0.955-0.970, n_D^{25} 1.472, [\alpha]_D^{25} -5.4 to -8.8°)$  on distillation yields 15% of hydrocarbons, chiefly cymene, 70% of ascaridole,  $b_a$  96-7°,  $d_{20}^{20} 0.9985, n_D^{25} 1.4769, <math>\alpha_D^{20} 0.7°$  and old samples only about 4% of d-camphor. Ascaridole explodes on contact with mineral acids; with FeSO<sub>4</sub> it yields isopropyl alc. and ascaridole glycol,  $C_{10}H_{18}O_4$ , m. 62.5-64, b. 271-2°,  $d_{20}^{20} 1.0981 n_D^{20} 1.4796$ . The Schotten-Baumann method yields the monobenzoate, prisms from warm alc., m. 136-7°. When heated to 150° with Bz<sub>2</sub>O the glycol yields the dibenzoate, white needles from alc., m. 116.5-7.5°. The glycol on oxidation with neutral KMnO<sub>4</sub> gives (a) ascaridic acid,  $C_{10}H_{10}O_5$ , slender needles from  $H_2O$ ,  $m_{11}16.5-117$ . Silver salt,  $C_{10}H_{14}O_4Ag_2$ . (b) Dibasic acid,  $C_{10}H_{16}O_4$ , brilliant rhombohedra, decomp. 186-7°. Silver salt. The structure of ascaridole is discussed; it is probably an unstable dioxide.

Catalysis in the Wet Way. SENDERENS. Chem. Ztg., 34, 940.—Alc. gives C<sub>2</sub>H<sub>4</sub> when catalyzed by Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, propanol gives propylene, butanol gives butylene. E. J. WITZEMANN.

Chlorinated Acetylene. José Rodríguez Mourelo and A. García Banús. Lab. quim. inorgán. orgán. escuela industrial, Madrid. Anales soc. espan. fis. quim., 9, 84-7.—Chloroacetylene may be obtained by heating at 40° under a reflux condener, alc. NaOH and 1,2-dichloroacetylene. A current of N is kept circulating thru the app. The product contains alc., dichloroacetylene, C<sub>2</sub>H, and aldehyde. This method of preparation is practically identical with that of Homann and Kirmreuther (cf. C. A., 4, 449).

H. S. Paine.

Action of Ammonia and of Sodium Carbonate on different Sugars in Dilute Aqueous Solution. Adolf Jolles. Vienna. Biochem. Z., 32, 97-100; see C. A., 5, 1904.—0.01 N aq. NH<sub>2</sub> did not affect dil. solns. of arabinose, dextrose, levulose, galactose or maltose, after 24 hrs., at 37°. The same conc. of aq. NaOH reduced the rotation to 0° or nearly 0°. The action of Na<sub>2</sub>CO<sub>3</sub> was also very slight as compared with that of NaOH.

F. J. BIRCHARD.

Action of Esters on Sodiated Benzyl Cyanide. F. Bodroux. Fac. sci., Poitiers. Bull. soc. chim., 9, 651-7; cf. C. A., 5, 1401.—The following is not reported in the earlier article: BzCHPhCN and BzCl give the compound BzOCPh: CPhCN, needles from ligroin, needles and lamellas from C<sub>0</sub>H<sub>0</sub>, m. 124-5°. PhCHNaCN and (CH<sub>2</sub>-CO<sub>2</sub>Et)<sub>2</sub> give 2,7-diphenyloctane-3,6-dione-1,8-dinitrile, (CH<sub>2</sub>COCHPhCN)<sub>2</sub>, lamellas, m. 156°. Chas. A. Rouller.

Hydroxyindazoles. III. P. FREUNDLER. Bull. soc. chim., 9, 657-61; cf. C. A., 5, 3232.—2-Benzeneazo-5-chlorobenzoic acid, orange-red needles, m. 126-7°; it is purified thru its barium sall, scales with H<sub>2</sub>O which it loses at 100°. 2-Benzeneazo-5-bromo-

bensoic acid, ruby-red prisms, m. 142-3°. The following azo acids were obtained by condensing the corresponding o-nitroso acids and amines in AcOH: PhN<sub>2</sub>C<sub>2</sub>H<sub>4</sub>CO<sub>2</sub>H; p-MeC<sub>2</sub>H<sub>4</sub>CO<sub>2</sub>H; p-ClC<sub>2</sub>H<sub>4</sub>N<sub>2</sub>C<sub>3</sub>H<sub>4</sub>CO<sub>2</sub>H; 2-p-tolueneazo-5-methylbensoic acid, red prisms, m. 122-5°, prepared from 5-methyl-2-nitrosobensoic acid, grayish prisms, m. 172-3° (decomp.); 2-p-olueneazo-5-chlorobenzoic acid, orange scales, m. 159-60°. Chas. A. ROUILLER.

Hydroxyindazoles. IV. Preparation of Hydroxyindazoles from Non-substituted Azo or Hydrazo Acids. P. Freundler. Bull. soc. chim., 9, 735-9; cf. C. A., 4, 1038.

—o-PhnHnHC<sub>0</sub>H<sub>4</sub>CO<sub>2</sub>H and its 5-chloro deriv. are converted into the corresponding phenylhydroxyindazoles by POCl<sub>2</sub> in pyridine. The 3,5-dichloro-7-hydroxyphenylindazole can be obtained from the dichloroazo acid by reduction with Zn dust and AcOH, while the azo and monochloroazo acids, under the same conditions, yield only the corresponding hydrazo acids.

Chas. A. Rouiller.

Hydroxyindazoles. P. Freundler. Compt. rend., 152, 1256-9; cf. preceding abstracts. Chas. A. Rouiller.

o-Benzeneazoxybenzoic Acid. P. FREUNDLER. Bull. soc. chim., 9, 739-41.—
o-Benzeneazoxybenzoic acid, PhN<sub>2</sub>OC<sub>2</sub>H<sub>4</sub>CO<sub>2</sub>H, yellowish white scales, is easily obtained by warming 42 g. PhNHOH with 55 g. o-ONC<sub>4</sub>H<sub>4</sub>CO<sub>2</sub>H in 500 cc. alc. Yield, 11-12 g.; the greater part of the reaction product consists of Ph<sub>2</sub>N<sub>2</sub>O and (HO<sub>2</sub>CC<sub>4</sub>H<sub>4</sub>)<sub>2</sub>N<sub>2</sub>O.

CHAS. A. ROUILLER.

Laws of Substitution in the Benzene Nucleus. A. F. Holleman. Bull. soc. chim., 9, I-XLV (July 20).—An address. Chas. A. Rouiller.

Reaction of Organomagnesium Compounds on Methyl Acetylpyrotartrate. Ph. Barbier and R. Locquin. Bull. soc. chim., 9, 717-22.—MeO<sub>2</sub>CCH<sub>2</sub>CHMeCO<sub>2</sub>Me gives, with Me<sub>2</sub>CHCH<sub>2</sub>MgBr and PhMgBr. the compounds Me<sub>2</sub>CHCH<sub>2</sub>CMe(OH)CMe-(CO<sub>2</sub>Me)CH<sub>2</sub>CO<sub>2</sub>Me, b<sub>14</sub> 157-8°, and HOCPhMeCMe(CO<sub>2</sub>Me)CH<sub>2</sub>CO<sub>2</sub>Me, oil, decompound distillation. The acids corresponding to these esters are unstable; efforts at hydrolysis led to the formation of HO<sub>2</sub>CCH<sub>2</sub>CHMeCO<sub>2</sub>H and of Me<sub>2</sub>CHCH<sub>2</sub>Ac and PhAc, resp.

Chas. A. Rouiller.

New Method for the Synthesis of Methyl Ketones. Ph. BARBIER AND R. LOCQUIN. Lab. chim. gén. fac. sci., Lyon. Bull. soc. chim., 9, 722-6.—PhCH<sub>2</sub>Ac was obtained in 40% yield by dropping slowly AcCMe<sub>2</sub>CO<sub>2</sub>Me into PhCH<sub>2</sub>MgCl in Et<sub>2</sub>O and hydrolyzing the resulting ester (cf. preceding abstr.) with KOH in MeOH.

CHAS. A. ROUILLER.

Reduction in the Presence of Finely Divided Palladium. PIERRE BRETEAU. Bull. soc. chim., 9, 729-32.—Phenanthrene is reduced over finely divided Pd at 150-60° to the tetra- and octohydro compds. Chas. A. ROUILLER.

Catalysis of Borneol and the Catalytic Reduction of Camphor. J. ALOY AND V. BRUSTIER. Bull. soc. chim., 9, 733-5.—Borneol vapors passed over reduced Cu at 300° are completely transformed into camphor; above 320° the amt. of camphor diminishes and at 420-30° terpenes are formed. Camphor alone at 130-220°, or in cyclohexanol at 180-200°, is not reduced by H and reduced Ni, but the oxime is easily reduced to bornyl-, dibornyl- and camphylamines, the secondary amine predominating. Chas. A. ROUILLER.

Constitution of Bixin. J. F. B. VAN HASSELT. Rotterdam. Rec. trav. chim., 30, 1-47.—Bixin, obtained by Zwick's method (Arch. Pharm., 238, 58) but freed from a brown amorphous impurity by washing by decantation with AcMe, large red needles from glacial AcOH, violet rhombs from AcOEt, mioroscopic crystals from amyl alc., violet, triclinic crystals from CHCl<sub>2</sub>, acute angle of the prism planes about 80°, n 1.50, strongly pleochroitic, m. 187°, has the comp. C<sub>22</sub>H<sub>24</sub>O<sub>5</sub> and not C<sub>22</sub>H<sub>24</sub>O<sub>5</sub>, as given

by previous workers. It dissolves in b. dil. KOH with loss of MeOH, forming norbixin,  $C_{20}H_{22}O_{3}$ , minute pale red, probably rhombic crystals, infusible, becomes colorless 240°, n 1.63. It is very easily oxidized and its comp. cannot be detd. by analysis; recourse was had to a detn. of the comp. of its Me derivs. Me<sub>x</sub>SO<sub>4</sub> easily reacts with it in KOH and MeOH, giving bixin, which, in turn, gives methylbixin,  $C_{20}H_{20}O_{4}$ , red, pleochroitic, triclinic rhombs, m. 156°, acute angle 68° 55′, n about 1.60. Zeisel detns. show that the 3 compds. contain 0, 1 and 2 OMe groups, resp. Methylbixin colors conc.  $H_{2}SO_{4}$  intensely blue, and KOH in EtOH easily hydrolyzes it to norbixin. Ethyl-

(2) bixin, MeO.C. HanO2.OEt, from bixin and Et.SO4, violet rhombs, m. 138°. When bixin is hydrolyzed in EtOH with KOH and then treated with Et.So., 1-ethylnorbixin, red needles with greenish reflection, m. 176°, and 1,2-diethylnorbixin, blue rhombs, m. 121°, are obtained. The mono-Et compd. is entirely similar in its properties to bixin and it is therefore assumed that the Et enters the same OH, designated (1), as is occupied by the Me in bixin; when methylated it gives 2-methyl-1-ethylnorbixin (ethylisobixin), red rhombs, m. 149°, isomeric with ethylbixin above, thus showing that the 2 OH groups in norbixin are not equiv. Similarly, when methylbixin is partially hydrolyzed with KOH in EtOH, isobixin, m. 178°, is obtained; it is sol. in but not hydrolyzed by aq. KOH. There facts indicate that the 1-OH group has weaker acid properties than the other. The salt hitherto described as di-K bixinate is really dipotassium norbixinate. 2-Potassium salt. Disodium salt, much more stable than the di-K salt. Dihydrobixin (cf. Marchlewski and Matejko, Bull. intern. acad. sci., Cracovie, 1905, 745) is C. Haio. Dihydromethylbixin, shining hexagonal lamellas, m. 174°. Dihydroisobixin, rhombs, m. 190°. Dihydronorbixin, rhombs, decomp. about 235°. The bixins add Br energetically. Decabromobixin, C20H24O2Br10, white powder; alks. remove all the Br. Decabromomethylbixin, white powder. The Hübl-Wijs method (Ber., 31, 750) showed that the bixins and their dihydro derivs. absorb the same amt. of halogens, indicating that the latter and H attack the mol. in different manners. Heated at 190° in H or some neutral solvent (Ph,NH) bixin loses 1 mol. m-Me<sub>2</sub>C<sub>2</sub>H<sub>4</sub>. The statement of Zwick that palmitic acid is formed by the action of superheated steam on bixin or of light on the Na salt is erroneous. The blue colors imparted to H.SO, by bixin, carotin and xanthophyll are very similar and it is possible that in all 3 cases substances with analogous chromophores are formed. The usual reagents for OH groups give negative results with bixin; the same is true for reagents for the CO group with methylbixin. With PhNHNH, in AcOH on the H4O bath bixin gives an amorphous orange product, m. 95°, having approx. the comp. of a phenylhydrazone, insol. in alk. Possibly it results from the oxidation of a -COH to a CO group; the author concludes that no CO group, as such, exists in bixin. Alk. KMnO<sub>4</sub> oxidizes 35.16% of the C of methylbixin to (CO<sub>2</sub>H)<sub>2</sub>, and 63.68% to CO<sub>2</sub>, while neutral KMnO<sub>4</sub> oxidizes 39.40 and 3.7% to CO<sub>2</sub> and (CO<sub>2</sub>H)<sub>2</sub>, resp. Other oxidizing agents give no definit results. The facts so far known as to the constitution of bixin may be represented by the formula m-Me<sub>2</sub>C<sub>2</sub>H<sub>4</sub>C<sub>18</sub>H<sub>19</sub>O<sub>3</sub>(COMe)CHOH. C. A. R.

Simultaneous Formation of Isomeric Substitution Products of Benzene. XV. A. F. HOLLEMAN. Rec. trav. chim., 30, 48-100; see following abstr. C. A. R.

Quantitative Investigation of the Introduction of a Halogen Atom into Phenol. I. J. RINKERS. Rec. trav. chim., 30, 48-100.—The % of p-BrC<sub>0</sub>H<sub>4</sub>OH in the bromination product of PhOH was detd. by means of the solidification p. curve of mixts. of the pure o- and p-compds., the Blanksma test (Chem. Weekblad, 1908, 93) having shown that none of the m-isomer was formed. To insure complete reaction and prevent the formation of polybromides, altho equiv. amts. of PhOH and Br were used, the Br was introduced either in vapor form dil. with air or CO<sub>2</sub>, or solvents (CS<sub>2</sub>, glacial)

AcOH, COL or HaO) were used. In the case of CS, the solvent was removed from the bromination product by simple distillation at 60°; with AcOH, NaHCO<sub>2</sub> was added and the BrC.H.OH extracted with Et.O; while in the case of CCl. NaOH was added, the CCl, removed by means of a separatory funnel and the BrC, H,OH extracted from the acidified soln. with Et.O. Pure o-BrC.H.OH, obtained by distillation of the dry picrate, m. 5.6°. The % of p-compd. formed were as follows: In CS<sub>3</sub>: -30°, 97.4; o°, 92.8; 30°, 86.2. In AcOH: 15°, 91.2; 30°, 87.5; 60°, 86.8. In CCl<sub>4</sub>: 30°, 88.6. Without solvent: 40°, 86.9; 60°, 90.2; 90°, 90.7; 131°, 81.6; 153°, 79.4; 180°, 77. In H<sub>2</sub>O, in AcOH at 90°, and without solvent at 0°, the results for various reasons (attack of the solvent by the Br, incomplete reaction, formation of dibromo compds.. etc.), were not quant. The results of the chlorination expts. without solvent were: 40°, 47.3; '90°, 50.2; and 155°, 48.8% p-ClC,H4OH. Pure o-ClC,H4OH solidifies 8.8°; m-compd., 32.8°; p-compd., 42.9°. The CI for the expts. was obtained from furning HCl and known weights of KMnO4. All attempts to obtain a quant. iodation of PhOH were unsuccessful. CHAS. A. ROUILLER.

Friedel and Crafts Reaction. XI. J. BÖESEKEN AND D. A. WITTOP KONING. Lab. chim. org. haute école techn., Delft. Rec. trav. chim., 30, 116-36.—The view previously accepted that the reaction between CaHa and SaCla was represented by the equation  $2C_1H_2 + S_2Cl_2 = Ph_2S + S + 2HCl$  (Rec. trav. chim., 24, 210) must be modified as some Ph.S. is always formed, either directly or by the action of S on Ph.S first formed. Attempts to determin this point by subjecting S and Ph.S. in the one case, and Ph.S alone, in the other, to the action of AlCl, were unsuccessful, for in the first case Ph.S. was formed, and in the second Ph.S and free S. Other reactions taking place are the following:  $Ph_sS_s + S = H_sS + (C_0H_sS)_s$  (thianthrene);  $2C_0H_s + 2S =$ H.S. + Ph.S.; C.H. + S = PhSH. The course of the reaction between 1 mol. S.Cl. with an excess of C.H., PhCl and PhMe in the presence of 1.25-1.5 mols. AlCl. was followed approx. quantitatively by detg. the amt. of S in the different products: that in H.S as Cu.S or by pptg. as Ag.S and detg. the Ag as AgCl; the free S by extracting the soln. of the reaction product with boiling Na<sub>2</sub>S of known conc. and detg. the S after extraction by oxidizing it to H<sub>2</sub>SO<sub>4</sub> with HNO<sub>5</sub>(d. 1.52); the S in disulfides by treating with conc. HNO, and weighing as Ba dinitrosulfonate. Each at. of free S represents an at. of S as monosulfide; that found as H<sub>x</sub>S is equiv. to an at. in thianthrene or in monosulfide (formed according to the equation  $2C_{1}H_{1} + 2S = Ph_{2}S + H_{2}S$ ); while the difference between the total S and that found as free S and HaS represents the Ph.S.. The results obtained are given below in ats. S (2 ats. S or 1 mol. S.Cl. being used in each expt.). The values are given in the order: Free S, H<sub>2</sub>S, monosulfide (+ thianthrene), S in side chain (in the case of PhMe), disulfide (by difference). C<sub>0</sub>H<sub>6</sub>: 0.781, 0.035, 0.816, -.--, 0.368. PhCl: 0.608-0.589, 0, 0.608-0.589, -.---, 0.784-0.822. PhMe: 0.295, 0.213, 0.508, 0.064, 0.920. That polysulfides are formed is made very improbable by the fact that in the case of CoHo the amt. of disulfide formed was found, by weighing as dinitrosulfonate, to be 0.360, in very close agreement with the value 0.368 found by difference. These results show that PhMe (and its sulfide) is more easily attacked by S than CaHe while PhCl is not attacked at all; substitution in the side chain is not marked; the reaction is complete only when all the S is combined as monosulfide and thianthrene. The amts. of thiophenol formed are very slight. CHAS. A. ROUILLER.

Action of Sulfur on Aromatic Sulfones. J. BÖESEKEN. Lab. chim. org. haute école techn., Delft. Rec. trav. chim., 30, 137-41.—While Ph<sub>2</sub>SO<sub>2</sub>, when heated with S, gives Ph<sub>2</sub>S almost quantitatively (Kraft and Vorster, Ber., 26, 2813), its p,p'-halogen and Me derivs. react in an entirely different manner, the former giving dihalogen substitution products of C<sub>2</sub>H<sub>2</sub> and the latter H<sub>2</sub>S. The nature and amts. of the prod-

ucts obtained by heating 10 g. of various sulfones at 200–360° with slightly more than 1 at. of S are given below. BrC<sub>2</sub>H<sub>4</sub>SO<sub>2</sub>Ph: unchanged sulfone, a little SO<sub>2</sub> and PhBr. (ClC<sub>2</sub>H<sub>4</sub>)<sub>2</sub>SO<sub>2</sub>: SO<sub>2</sub>, 3 g. C<sub>2</sub>H<sub>4</sub>Cl<sub>2</sub>. ClC<sub>2</sub>H<sub>4</sub>SO<sub>2</sub>C<sub>2</sub>H<sub>4</sub>Br: SO<sub>2</sub>, 2.25 g. C<sub>2</sub>H<sub>4</sub>ClBr, some C<sub>2</sub>H<sub>4</sub>Br<sub>2</sub>. (BrC<sub>2</sub>H<sub>4</sub>)<sub>2</sub>SO<sub>2</sub>: SO<sub>2</sub>, 1 g. C<sub>2</sub>H<sub>4</sub>Br<sub>2</sub>. MeC<sub>2</sub>H<sub>4</sub>SO<sub>2</sub>C<sub>2</sub>H<sub>4</sub>Br: (240–50°) H<sub>2</sub>S, H<sub>2</sub>O, HBr and a red oil; (340°) traces of HBr, SO<sub>2</sub>, C<sub>3</sub>H<sub>4</sub>Br<sub>2</sub>; (340° with C instead of S) practically unchanged. At 360°, (BrC<sub>2</sub>H<sub>4</sub>)<sub>2</sub>SO<sub>2</sub> suffers total decomp. with S as well as with Cu; does not react with Cu below 320°; and gives traces of SO<sub>2</sub> and C<sub>2</sub>H<sub>3</sub>Br with C at 320°. Chas. A. Rouiller.

Action of Perhydrol on  $\alpha$ -Diketones. J. BÖESEKEN. Lab. chim. org. haute école techn., Delft. Rec. trav. chim., 30, 142-7.—The poor yields of Me<sub>2</sub>CCO<sub>2</sub>H obtained by the action of H<sub>2</sub>O<sub>3</sub> on Me<sub>2</sub>CCOCO<sub>2</sub>H is due to partial oxidation of one of the Me groups, with evolution of CO<sub>2</sub> and formation of Me<sub>2</sub>CHCO<sub>2</sub>H. Similarly 2,4,6-Me<sub>2</sub>-C<sub>2</sub>H<sub>2</sub>COCO<sub>2</sub>H yielded a substance giving the reactions of o-C<sub>2</sub>H<sub>4</sub>(CO<sub>2</sub>H)<sub>2</sub>. On the other hand, p-ClC<sub>2</sub>H<sub>4</sub>COCO<sub>2</sub>H reacts normally. Ac<sub>2</sub> gives only AcOH, no CO<sub>2</sub> being evolved. Heated 2 hrs. on the H<sub>2</sub>O bath with excess of H<sub>2</sub>O<sub>2</sub>, (CO<sub>2</sub>H)<sub>2</sub>, (CONH<sub>2</sub>)<sub>2</sub>, (CONHPh)<sub>3</sub> and HO<sub>2</sub>CCONHPh gave 65.6, 1.0, 1.5 and 1.6% CO<sub>3</sub>, resp.; OH groups therefore protect the CO of  $\alpha$ -diketones from oxidation to CO<sub>2</sub>H, but it is N especially which has this protective influence. Thus, isatin is not attacked by H<sub>2</sub>O<sub>2</sub>, while  $\beta$ -naphthoquinone (containing the grouping —CCOCOC—) is easily converted into o-HO<sub>2</sub>CC<sub>2</sub>H<sub>4</sub>CH: CHCO<sub>2</sub>H and stearoxylic acid gives pelargonic and azelaic acids quant. C. A. R.

Friedel and Crafts Reaction. J. BÖRSEKEN. Lab. chim. org. haute école techn., Delft. Rec. trav. chim., 30, 148-50.—C<sub>3</sub>Cl<sub>4</sub> and CHCl<sub>5</sub> combine, in the presence of even small amts. of AlCl<sub>5</sub>, giving CCl<sub>2</sub>CCl<sub>2</sub>CHCl<sub>5</sub>, the slight losses of AlCl<sub>5</sub> observed being due to resinifications caused by impurities in the com. product. The reaction is reversible, the C<sub>5</sub>HCl<sub>7</sub> decomp. gradually into its components at 121°. The same is true for the systems C<sub>2</sub>HCl<sub>2</sub>-CHCl<sub>3</sub>, C<sub>2</sub>HCl<sub>3</sub>-CCl<sub>4</sub>, C<sub>2</sub>H<sub>2</sub>Cl<sub>2</sub>-CCl<sub>4</sub>, and C<sub>2</sub>H<sub>3</sub>Cl<sub>2</sub>-CHCl<sub>3</sub>. According to the authors, this solves the question of the Friedel-Crafts reaction: in order to secure condensation, an unsatd. compd. and one which can be so activated that it can combine with the unsatd. compd. must be brought together in the presence of a catalyzer, the reaction being made possible by the loss of free energy. In the case of unsatd. ring compds. in general, the reaction does not stop at this point; the system, having less free energy than its dihydro deriv., tends to re-establish itself with evolution of HCl; this loss of free energy is doubtless so great that the primary addition product is too unstable to permit of its isolation.

C. A. R.

Derivatives of Diaminoacetone. A. P. N. Franchimont and J. V. Dubsky. Leyden. Rec. trav. chim., 30, 177-82; cf. Ibid., 26, 223.—Acetonediethylurethan, CO(CH<sub>2</sub>NHCO<sub>2</sub>Et)<sub>2</sub>, from CO(CH<sub>2</sub>NH<sub>2</sub>HCl)<sub>2</sub> and ClCO<sub>2</sub>Et in aq. alc. in the presence of Na<sub>2</sub>CO<sub>3</sub>, gleaming scales, m. 136-7°; it is unaffected by boiling 2.5 hrs. with a large excess (60 parts) of Ac<sub>2</sub>O, but if a little ZnCl<sub>2</sub> is added, acetonediethylacetylurethan, CO(CH<sub>2</sub>NAcCO<sub>2</sub>Et)<sub>2</sub>, needles, m. 61-2°, is obtained. Acetonediethylnitrourethan, from the ethylurethan and 10 parts abs. HNO<sub>3</sub>, silky needles, m. 56-7°. Tetracetyldiaminoacetone, obtained by b. CO(CH<sub>2</sub>NH<sub>2</sub>)<sub>2</sub> along time with Ac<sub>2</sub>O in large excess, needles, softens 98°, m. 108°.

Acetylation of Substituted Acetamides. A. P. N. Franchimont and J. V. Dubsky. Rec. trav. chim., 30, 183-5.—AcNHMe and AcNHEt are converted into the corresponding di-Ac compds. by long boiling with 7 parts of Ac<sub>2</sub>O, while (CH<sub>2</sub>NHAc)<sub>2</sub>, m. 175°, gives tetracetylethylenediamine, glassy crystals, m. 156-7°. C. A. ROUILLER.

Products of the Reaction between Diaminoacetone Hydrochloride and Potassium Isocyanate. "Amino- and Ureomethylethyleneurein." A. P. N. Franchimont and J. V. Dubsky. Leyden. *Rec. trav. chim.*, 30, 186–203; see C. A., 5, 1905.

CHAS. A. ROUILLER.

Bromo-Sodio Amides and their Role in the Hofmann Transposition. CH. MAUGUIN. Ann. chim. phys., 22, 297-369; cf. C. A., 4, 583.—AcNHBr, orthorhombic, hemimorphic crystals with 1 H<sub>2</sub>O, pe' 34° 50', 2E 127°, no-no 0.064 (parallel to h'), m<sub>a</sub>—n<sub>m</sub> 0.030. It loses H<sub>2</sub>O at 50°, giving needles m. 106°. AcNHCl, from AcNH<sub>2</sub>, o. 5 mol. ZnO and 2 ats. Cl, anhydrous, mica-like lamellas, m. 110°. Propionicbromoamide, needles, m. 80°. Chloroamide, m. 34°. Isobutyricbromoamide, needles, m. 92°, from Me<sub>2</sub>CHCONH<sub>2</sub>, Br and NaOH in CHCl<sub>3</sub>. BzNHBr, monoclinic crystals, with perfect cleavage along g; ph' 80° 42', mm (on h) 37° 4', 2E 115°; optically negative. Silver aceticbromoamide, from a fresh soln. of AcNNaBr and AgNO3, white ppt., very explosive when dry, sol. completely in HNO3; also sol. in NH3, but soon there is evolution of N and abundant pptn. of AgBr. Sodium aceticchloroamide; silver salt. The Na salts, on heating alone or in Et<sub>2</sub>O, decomp. with formation of NaBr and an isocyanate. Isopropyl isocyanate, from Me<sub>2</sub>CHI and AgNCO, b. 70-5°; with NH<sub>2</sub> it forms isopropylurea, needles, m. 154°; with PhNH2, phenylisopropylurea, needles, m. 156°. With abs. EtOH, the above Na bromoamides give Et alkylcarbamates. MeNH-CO<sub>2</sub>Et, b<sub>18</sub> 80°, b. 170°, d<sub>18</sub> 1.035, n<sub>D</sub> 1.421. EtNHCO<sub>2</sub>Et, b<sub>18</sub> 79-80°, b. 170°, d<sub>18</sub> 0.997, np 1.423. Ethyl isopropylcarbamate, b<sub>18</sub> 79°, d<sub>18</sub> 0.957, np 1.424. With urethans the Na bromoamides give alkylureocarbonate (allophanic) esters. Ethyl methylwreocarbonate, MeNHCONHCO2Et, needles, m. 134°. Methyl ester, m. 163°. Propyl ester, m. 130°. Methyl ethylureocarbonate, needles, sublimes 40-50°, m. 95°. Ethyl ester, needles, m. 72°. Propyl ester, birefractive lamellas, m. 81°. Isobutyl ester, birefractive lamellas, m. 87°. Isoamyl ester, needles, m. 67-8°. Methyl isopropylureocarbonate, m. 70°, monoclinic prisms, ph'(001)(100) 64° 27', a:b:c=0.697: 1: 1.115. Ethyl ester, m. 40°. With amides, the Na bromoamides give acylamides. MeNHCONHAc, m. 180°, triclinic crystals, a:b:c=1.483:1:0.9953. If the amide and bromoamide contain different acyl radicals, 3 products are formed; thus, AcNNaBr and EtCONH, give MeNHCONHAc, EtNHCONHAc and EtNHCONHCOEt. EtNHCONHAC, m. 124°, triclinic, lozenge-shaped tables, easy cleavage along h', quite easy along p and g'; groups of 2 crystals joined along p, the faces h' being in the same plane, are often observed. Acetylpropylurea, micaceous amellas, m. 115°. PrNHCONHCOPr, scales, m. 100°, triclinic, flattened lozenges, a:b:c=1.245: 1: 0.5054. AcNHBr, boiled a few mins. with 1 mol KOH and 1 mol. EtO<sub>2</sub>CCONH<sub>2</sub>, gives EtO<sub>2</sub>CNH<sub>2</sub>, EtO<sub>2</sub>CNHCONH<sub>2</sub> (in small amt.), KO<sub>2</sub>CCONHCONHCO<sub>2</sub>Et and a number of other products (Br, KBr, AcNH2, etc.). Potassium carboxyethyloxaloureide, more easily obtained by adding 1 mol. EtO<sub>2</sub>CCONH<sub>2</sub> to 1 mol. AcNH<sub>2</sub> and 2 ats. Br in 2 mols. KOH and 500 cc. H<sub>2</sub>O and warming 0.5 hr. at 30°, microscopic, felted needles. Copper salt, bluish green needles, Silver salt, white ppt. All these salts are easily hydrolyzed. Boiling H<sub>2</sub>O decomp. the K salt into EtO<sub>2</sub>CNHCONH, and HO<sub>2</sub>CCO<sub>2</sub>K; NH, into EtO<sub>2</sub>CNHCONH<sub>2</sub> and KO<sub>2</sub>CCONH<sub>2</sub>.H<sub>2</sub>O; EtNH<sub>2</sub> into EtO<sub>2</sub>CNHCONH<sub>2</sub> and KO2CCONHEt. Ethyl carboxyethyloxaloureide, needles, m. 149°, is obtained either from EtI and the Ag salt above, or from EtO, CNHCONH, and ClCOCO, Et. H<sub>2</sub>O, EtOH, NH<sub>2</sub> and EtNH<sub>2</sub> decomp. it with formation of EtO<sub>2</sub>CNHCONH<sub>2</sub> and (in the last 2 cases) (CONH<sub>2</sub>)<sub>2</sub> and (CONHEt)<sub>2</sub>, resp. In the prep. of the above ureide, the EtO2CNH2 is extracted with Et2O, which at the same time takes out the free Br, the latter in the presence of H<sub>2</sub>O, giving, with Et<sub>2</sub>O, CH<sub>2</sub>B CHO, which condenses with 2 mols. EtO2CNH2, forming bromoethylidinediurethan, CH2BrCH(NHCO2Et)2, needles, m. 146°. AcNNaBr and CH<sub>2</sub>(CO<sub>2</sub>Et)<sub>2</sub> in Et<sub>2</sub>O, give AcNH<sub>2</sub>, a little MeNHCONHAC and (EtO<sub>2</sub>C)<sub>2</sub>C: C(CO<sub>2</sub>Et)<sub>2</sub>. CHAS. A. ROUILLER.

o-Dibenzoylbenzene and its Homologs. A. Guyot and F. Vallette. Ann. chim. phys., 23, 363-97; cf. Bull. soc. chim., 35, 1124; C. A., 4, 1486.—α-Tolyl-α'-phenyl-α'-hydroxy-β,β'-benzo-α,α'-dihydrofurfurane (I), from PhMgBr and excess

(about 1.5 mol.) of tolylphthalide, cryst. powder, slowly loses  $H_2O$  at room temp., more rapidly on warming, giving  $\alpha'$ -phenyl- $\alpha$ -tolyl- $\beta$ ,  $\beta'$ -bensofurfurane (II), also obtained by adding a few drops conc. HCl to a boiling alc. soln. of the above compd.,

yellow needles, m. 83°. The same compd. is obtained by dehydration of a-phenyl-a' $tolyl-\alpha'-hydroxy-\beta,\beta'-benzo-\alpha,\alpha'-dihydrofurjurane$ , which could not be obtained in cryst. form. The yellow, fluorescent solns. of the dehydration product are slowly decolorized in air, instantly by Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, with formation of o-benzoyltoluylbenzene, prisms, m. 139°, sol. in conc. H,SO4 with intense fuchsin-red color. With N,H4.H4O, it gives phenyltolylphthalazine (III), white scales, m. 117°, while with excess of Na-Hg it forms o-benzhydryltolhydrylbenzene, HOCHPhC,H,CH(OH)C,H,Me, scales, m. 104°. It can be obtained directly from the dihydrofurfurane (I), while the compd. (II) is reduced to  $\alpha'$ -phenyl- $\alpha$ -tolyl- $\beta$ ,  $\beta'$ -benzo- $\alpha$ ,  $\alpha'$ -dikydrojurfurane, needles, m. 104°. With a large excess of PhMgBr, tolylphthalide gives o-tolhydryltriphenylcarbinol, HOCPh,CaH,CH-(OH)C<sub>6</sub>H<sub>4</sub>Me, m. 182°; heated in C<sub>6</sub>H<sub>6</sub> in the presence of conc. H<sub>2</sub>SO<sub>4</sub> it loses 2 mols. H.O. forming 10-phenyl-q-tolylanthracene, slightly yellow prisms, m. 192°, easily oxidized to 10-phenyl-9-tolyl-9,10-dioldihydroanthracene, needles with EtOH, effloresces in air and then m. 212°, sol. in conc. H,SO4 with intense indigo-blue color; it has strong oxidizing powers. The anthracene was also synthesized by treating phenylethoxyanthranol with MeC\_H\_MgBr and reducing the resulting compd., probably 10-tolyl-9phenyl-10-hydroxy-9-ethoxydihydroanthracene. m. 199°, with Zn and HCl. When HCl in glacial ACOH instead of H.SO4 is used to dehydrate the carbinol, only 1 mol. H.O is lost, the product being  $\alpha$ -tolyl- $\alpha'$ ,  $\alpha'$ -diphenyl- $\beta$ ,  $\beta'$ -benzo- $\alpha$ ,  $\alpha'$ -dihydrofurfurane, needles, m. 123°; on oxidation it gives α-tolyl-α', α'-diphenyl-α-hydroxy-β,β'-benzo-α, α'-dihydrofurfurane, m. 130°, which can also be obtained from MeC\_H\_MgBr and diphenylphthalide,  $\alpha, \alpha'$ -Ditolyl- $\alpha'$ -hydroxy- $\beta, \beta'$ -venzo- $\alpha, \alpha'$ -dihydrofurfurane, from tolylphthalide and  $\rho$ -MeC<sub>2</sub>H<sub>4</sub>MgBr, very easily dehydrated.  $\alpha, \alpha'$ -Ditolyl- $\beta, \beta'$ -benzofurfurane, yellow needles, m. 125°, oxidized quantitatively by CrO<sub>2</sub> to C<sub>2</sub>H<sub>4</sub>(COC<sub>2</sub>H<sub>4</sub>Me)<sub>3</sub>. Ditolylphthalazine, needles, m. 221°. Ditoluytbenzene phenylhydrazone, MeC.H.COC.H.C. (C.H.Me): NNHPh, yellow prisms, m. 171°. o-Ditolhydrylbensene, scales, m. 118°. α,α'-Ditolyl-β,β'-benzo-α,α'-dikydrofurfurane. needles, m. 89°. o-Tolkydrylphenylditolylcarbinol, prisms, m. 138°. 2-Methyl-9,10-di-p-tolylanthracene, yellow prisms. 2-Methyl-9,10-di-p-tolyl-9,10-dioldihydroanthracene, prisms with EtOH, effloresces in the air and m. 207°. o-Benzoylnaphthoylbensene, obtained by oxidizing the oily product of the reaction between α-C<sub>10</sub>H<sub>7</sub>MgBr and phenylphthalide, m. 104°. o-Tolwylnaphthoylbenzene, prisms, m. 144°. CHAS. A. ROUILLER.

 $\alpha, \alpha$ -Dialkyl- $\beta$ -keto Alcohols. III. E. E. BLAISE AND I. HERMAN. Ann. chim. phys., 23, 522-44; cf. C. A., 4, 763, 2451.— $\beta$ -Acetoxypseudobutyl phenyl ketone, AcOCH<sub>1</sub>-CMe<sub>1</sub>Bz, from PhZnBr and AcOCH<sub>2</sub>CMe<sub>2</sub>COCl, b<sub>11</sub> 155.5-6.5°. Yield, 70%. p-Nitrophenylhydrazone, orange-yellow needles, m. 125°. Cold 10% KOH saponifies the acetoxy compd. to  $\beta$ -hydroxypseudobutyl phenyl ketone, b<sub>12</sub> 152-3°. Oxime, needles, m. 122.5°. p-Nitrophenylhydrazone, yellow needles, m. 157°. Phenylurethan, needles, m. 89°. Hot alk. decomp. the above ketones into HCHO and Me<sub>2</sub>CHBz. Heated with P<sub>4</sub>O<sub>10</sub> to 200°, the hydroxy compd. gives benzoylmethyltrimethylene(?), (CH<sub>2</sub>): CMeBz, b<sub>10</sub> 117-9°; p-nitrophenylhydrazone, yellow cryst. powder, m. 175°. It is isomeric with tigylbenzene, MeCH: CMeBz, b<sub>10</sub> 117.5°, p-nitroph nylhydrazone, reddish yellow needles, m. 136°, and with dimethylacrylbenzene, Me<sub>2</sub>C: CHBz, b<sub>10</sub> 117°, p-nitrophenylhydrazone, m. 132°. These 2 compds. are obtained from PhZnBr and the cor-

responding acid chlorides. HO<sub>2</sub>CCMe<sub>2</sub>CHPhOAc is obtained in 33-5% yield by b. HO<sub>2</sub>CCMe<sub>2</sub>CHPhOH 1 hr. with 2 mols. AcCl, decomposing the excess of AcCl with H<sub>2</sub>O, extracting with Et<sub>2</sub>O, then with 25% K<sub>2</sub>CO<sub>3</sub>, and pptg. with conc. HCl. Phenylacetoxypivalyl chloride, from the acid and SOCl<sub>2</sub>, m. 41°, b, 151°. p-Toluide, m. 191-2°. With EtZnI the chloride gives a compound b<sub>10</sub> 118.5-9.5° which does not give the ketone reactions and was not identified, β-phenyl-β-acetoxypseudobutyl ethyl ketone, m. 42°, b<sub>11</sub> 160-5°, and isobutenylbenzene, Me<sub>2</sub>C: CHPh, b<sub>10</sub> 63.5°, whose nitrosite m. 154° (cf. Angeli, Ber., 25, 1962, who gives 112° as the m. p.). Attempts to saponify the acetoxy ketone result either in its decomp. into BzH and Me<sub>2</sub>CHCOEt or it is unaffected. Dimethylatropyl chloride, Me<sub>2</sub>C: CPhCOCl, b<sub>11</sub> 120.5°. p-Toluide, needles, m. 135-6°. Dimethylatropyl ethyl ketone, b<sub>13</sub> 124°; p-nitrophenylhydrazone, gleaming, reddish yellow needles, m. 129°. Chas. A. ROUILLER.

Condensation of Ethyl Acetate with its Higher Homologs. A. Wahl. Ann. chim. phys., 23, 545-55; cf. C. A., 5, 1590. Chas. A. Rouiller.

Nitropyrrole. A. Angeli and Luigi Alessandri. Lab. chim. farm. r. ist. studi sup., Firenze. Atti accad. Lincei, 20, I, 311-4.—Nitropyrrole, yellowish scales or prisms from ligroin, light yellow, opaque rhombohedrons from  $H_2O$ , is obtained by treating pyrrole in  $Et_2O$  with 1 at. Na wire and 1 mol.  $EtNO_2$ , adding ice after several days, extracting with  $Et_2O$ , adding excess of AgNO<sub>2</sub> to the brown aq. soln., washing the pptd. Ag salt free from AgNO<sub>2</sub>, adding NaCl soln. to the still moist ppt., passing  $CO_2$  through the resulting soln. and extracting with  $Et_2O$ . The NO<sub>2</sub> probably enters the  $\beta$ -position. Nitro- $\alpha$ , $\beta$ '-dimethylpyrrole, prisms, m. 111°, is obtained similarly, but as the Na salt is not very sol. in  $H_2O$ , it can be sepd. from the NaNO<sub>2</sub> without previously converting it into the Ag salt. These compds. are very stable in alc. to KMnO<sub>4</sub>.

Decacyclene and its Alleged Power to Dissolve Graphite. M. Padoa. Lab. chim. gen. r. univ. Bologna. Atti accad. Lincsi, 20, I, 345-7; cf. Ostromisslenskii, C. A., 2, 656.—Decacyclene is obtained in much better yield (12 g. per 100 g. of acenaphthene) by working with sealed tubes; 17 g. acenaphthene and 4 g. of S are heated 2 hrs. at 200°, the tubes cautiously opened to relieve the pressure due to H<sub>2</sub>S, heated 1 hr. longer at 250°, the contents extracted 4-5 times with b. PhMe and the decacyclene cryst. from PhNO<sub>2</sub>; m. 389.5°. Expts. with 3 graphites (black, friable, opaque blocks from Bavaria, gleaming, unctuous lamellas from Ceylon, hard, opaque Siberian graphite) showed no indication of solubility in the decacyclene; the f. p. was unchanged and the graphite was recovered unaltered. Chas. A. Rouiller.

New Oxidation of α-Methylindole. G. PLANCHER AND U. COLACICCHI. Ist. chim. gen. r. univ. Parma. Atti accad. Lincei, 20, I, 453-7.—H<sub>2</sub>O<sub>2</sub> in Et<sub>2</sub>O (15%) has been found to be a very useful oxidizing reagent. With it methylindole gives a compound C<sub>18</sub>H<sub>16</sub>ON<sub>2</sub>, m. 209-10°, almost insol. im most org. solvents, does not give the Angeli indole reaction, gives a red color with b. glacial AcOH, dissolves in conc. H<sub>2</sub>SO<sub>4</sub> with intense blue color and H<sub>2</sub>O ppts. green flocks, is unchanged by conc. or alc. KOH. Reduced with Sn and HCl it forms dihydromethylketole. The same compd. is obtained by the use of Caro's reagent. It probably has the constitution (I) or (II), the first being more likely the correct one.

CHAS. A. ROUILLER.

N-Phonyl Esters of Oximes. A. Angeli, L. Alessandri and M. Aiazzi-Mancini.

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Lab. chim. farm. r. ist. studi sup. Firenze. Atti accad. Lincei, 20, I, 546-55.—
N-Phenylbenzaldoxime and EtMgI form bhenyl-α-phenylpropylhydroxylamine, EtCHPhNPhOH, prisms, m. 127°, turms yellow in the air, gives PhNO with KMnO<sub>4</sub> and a
yellow oi! with other oxidizing reagents. With PhMgBr instead of EtMgI, diphenylmethylphenylhydroxylamine, m. 127°, is obtained on treatment with yellow HgO,
H<sub>2</sub>O<sub>3</sub>, long boiling with Grignard reagents or BzH, it is oxidized to a compound C<sub>19</sub>H<sub>10</sub>ON,
light yellow refractive scales, m. 214°; while Zn and AcOH reduce it to Ph<sub>2</sub>CHNHPh.
CrO<sub>2</sub> oxidizes it, as well as the compd. m. 214°, to PhNO and BzPh. N-Phenylbenzaldoxime is reduced by Zn and NH<sub>4</sub>Cl to PhCH: NPh. N-Benzylbenzaldoxime,
which can be obtained by the oxidation of Ph<sub>2</sub>NOH with HgO, gives with PhMgBr
diphenylmethylbenzylhydroxylamine, m. 105°; HgO oxidizes it to a compound C<sub>20</sub>H<sub>17</sub>ON,
m. 159°, which, with CrO<sub>2</sub>, gives BzH and BzPh. These facts confirm the authors'
views (C. A., 4, 1739) that the N-substitution products of oximes have the constitution RCH: NR: O.

Relations between some Derivatives of Oxygen and Nitrogen. A. ANGELI. Atti accad. Lincei, 20, I, 625-7.—Continuation of C. A., 5, 94. Chas. A. ROUILLER.

Preparation and Phototropy of some Osazones. II. M. PADOA AND L. SANTI. Lab. chim. gen. r., Univ. Bologna. Atta accad. Lincei, 20, I, 675-80; cf. C. A., 5, 277.—The following osazones, unless otherwise stated, are phototropic. Bensil-mtolylosazone, canary-yellow needles, m. 163°. Piperyl-m-tolylosazone, minute yellow needles, m. 187°, turns brick-red in the sun and is decolorized at 130-5°. Anisil-mtolylosazone, light yellow scales, m. 150.5°, assumes in the sunlight a bright orange color which disappears at about 80°. Piperyl-β-naphthylhydrazone, obtained in small amt. in the prep. of the osazone, yellow cryst. powder, m. 162°; it could not be detd. with certainty if it is phototropic. The osazone seps. from CHCl, with 1 mol. of solvent in yellow, non-phototropic crystals, m. 80°, solidifies and m. again at the same temp. as the osazone (207°). Anisil-β-naphthyiosazone, pale yellow needles in stars from CaHa-EtOH, m. 165-9°, assumes a wine-red color in sunlight which disappears at 55°; from C<sub>2</sub>H<sub>2</sub> alone it sep. with 1 mol. of solvent as a non-phototropic, white powder, m. 155-8°. Anisil-o-tolylosazone, non-phototropic orange-yellow crystals, m. 168°. Anisil-p-tolylosazone, light yellow powder. m. 153°, assumes a red color in the sunlight which disappears at about 85°; benzene addition product, white, silky, non-photo-CHAS., A. ROUILLER. tropic needles, m. 166°.

Isomeric Tanacetyl Alcohols. V. Faolini. Ist. chim.-farm. r. univ. Roma. Atti accad. Lincei, 20, I, 765–9.—Through the acid phthalate (cf. Semmler, "Die aetherische Oele," III, 134, 137) the author has succeeded in sepg. from the alc. obtained from tansy oil a pure d-tanacetyl or  $\beta$ -thugyl alc. and an impure  $\alpha$ -thugyl alc. Cf. following abstr. for the experimental data.

C. A. ROUILLER.

Derivatives of a d-Tanacetyl Alcohci. V. Paolini. Atti accad. Lincei, 20, I, 769-72; cf. preceding abstr.—Starting with a tanacetyl alc. b. 206-9°,  $d_{20}$  0.925,  $m_{D}$  1.4635,  $[\alpha]_{D}$  69° 49′, boiling it in petroleum ether 12-16 hrs. with a slight excess of Na, dropping it into the calc. amt. of phthalic anhydride, in petroleum ether, letting stand 48 hrs., adding an equal vol. of  $H_{2}O$ , pptg., after 24 hrs., the solid formed several times from  $C_{0}H_{0}$  by means of petroleum ether and finally crystallizing from  $C_{0}H_{0}$  pure  $\beta$ -thujyl hydrogen phthalate, is obtained in needles m. 120°,  $[\alpha]_{D}$  91° 27′. Silver salt, white ppt., m. 85-6°. Calcium salt, white downy needles. Strychnine salt, needles, m. 177-8°,  $[\alpha]_{D}$  36° 78′. With alc. KOH the acid ester gives  $\beta$ -thujyl alcohol, b. 206°, d. 0.9229,  $m_{D}^{16}$  1.4625,  $[\alpha]_{D}$  114° 67′. Treated with  $C_{0}H_{4}(CO)_{2}O$  it gives directly the pure phthalate m. 120°, and the ketone resulting from its oxidation with CrO<sub>3</sub> is pure  $\beta$ -thujone.

Hydrazones. R. Ciusa and L. Vecchiotti. Ist. chim. gen., univ. Bologna, Atti accad. Lincei, 20, I, 803-7; cf. C. A., 4, 2454.—Of the following hydrazones, all but 1 were obtained in 2 chromoisomeric forms (in 1 case in 3 forms). (In the following formulas R = \$\rho\_0.NC.H.NHN:) PhCH: R; crude product, brownish yellow, flattened needles; orange needles from boiling alc. but yellow scales if pptd. from the hot soln. with H<sub>2</sub>O and red crystals if pptd. from cold soln. The red passes into the yellow form at 140°; all m. 194-5°. On crystn. from various solvents the red and yellow forms give the orange modification, the red first passing into the yellow as soon as it comes in contact with the hot solvent, more slowly in cold solvents. The red form alone sep. from HCONH<sub>2</sub>. o-O<sub>2</sub>NC<sub>2</sub>H<sub>4</sub>CH:R; orange-red needles from glacial AcOH, becomes distinctly red at 190°, and when pptd. from hot or cold alc. with H<sub>2</sub>O it gives an orange-yellow form, going back into the red when it sep. from AcOH. Both m. 250-1°. m-O,NC,H,CH: R; orange-red from AcOH, m. 250-1°, pptd. by H,O from alc. in a yellow form, m. 248°, which passes back into the red when crystallized from AcOH. When heated at 130° the yellow form passes thru orange-red to red, the reverse change taking place as it cools. p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH: R; brick-red scales, m. 247°, from AcOH, orange-yellow from alc., m. 245°. Anisal-p-nitrophenylhydrazone is obtained in only 1 form, reddish violet needles, m. 160°. CHAS. A. ROUILLER.

Structure of Azoxy Compounds. A. Angeli and Luigi Alessandri. R. ist. studi sup., Firenze. Atti accad. Lincei, 20, I, 896–900.—p-Nitroazoxybenzene (Zinin, Ann., 114, 218), m. 152°, light yellow microscopic needles grouped into leaves. p-Nitroazobenzene, from PhNO and  $H_2NC_6H_4NO_2$ , red laminas, m. 135°, when treated in AcOH with  $H_2O_2$ , gives a compd. similar to Zinin's but nevertheless differing from it in some respects; m. 148°, is less sol. in various solvents and sep. in large gleaming prisms. With all caution the authors advance the opinion already expressed by some that azoxy compds. have not the structure usually assigned to them and that the above 2 products have the formulas O: NPh:  $NC_0H_4NO_2$  and PhN:  $N(:O)C_0H_4NO_2$ .

CHAS. A. ROUILLER.

Crystallographic Study of Monoethyl p-Tolylisosuccinamate. Aristide Rosati. Ist. min. r. univ. Roma. Atti accad. Lincei, 20, I, 921-2.—Et p-tolylisosuccinamate,  $C_{18}H_{17}O_2N$  (Comanducci and Lobello, Rend. accad. sci. fis. math. Napoli, Jan., 1905) monoclinic, tabular crystals, a:b:c=3.4327:1:1.4716,  $\beta$  68° 49′, (100), (001), (110), (111), perfect cleavage parallel to (001). Chas. A. Rouiller.

Dehydration of the Glycols from Anethole and Isosafrole. V. PAOLINI. Attiaccad. Lincei, 20, I, 940-6.—The  $\beta$ -glycol derived from anethole, when dehydrated with 20%  $H_2SO_4$  on the  $H_2O$  bath, gives a pure ketone (Tiffeneau and Daufresne, C. A., 1, 2574) which does not show the Angeli aldehyde reaction. The same is true of the glycol from isosafrole, but if the dehydration is carried out under the conditions employed by Balbiano and Paolini (Gazz. chim. ital., 36, 291), viz., by heating with ZnCl<sub>2</sub> at 130°, or if the glycol is b. a long time with  $H_2SO_4$ , altho the product is chiefly the ketone, yet a part of it does give the Angeli reaction. Balbiano's assertion that the product consists exclusively of aldehyde (C. A., 4, 1617) is therefore erroneous.

CHAS. A. ROUILLER.

Derivatives of Hydroxyhydroquinone. IV. G. BARGELLINI AND ERMANNO MARTEGIANI. Ist. chim. r., Univ. Roma. Atti accod. Lincei, 20, II, 18-25; cf. C. A., 5, 2098.—In the prep. of 2,4,5-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>2</sub>COEt, there is formed, besides 24 g. of the trimethyl compd. from 30 g. (MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, 11 g. of a dimethyl compd. which is probably 4,5-dimethoxy-2-hydroxypropiophenone, needles, softens 120°, m. 124-6°, gives a green color with aq. FeCl<sub>2</sub> and a greenish yellow color with conc. H<sub>2</sub>SO<sub>4</sub>. Acetyl derivative, needles, m. 117-8°. Benzoyl derivative, woolly needles, m. 110-1°. With Me<sub>2</sub>SO<sub>4</sub> it gives (MeO)<sub>2</sub>C<sub>6</sub>H<sub>2</sub>COEt. 2,4,5-Trimethoxypropiophenone phenylhydrazone,

m. 113°. Treated with amyl nitrite and HCl, in amyl alc., the trimethyl compd. gives an isonitroso derivative, (MeO)<sub>2</sub>C<sub>2</sub>H<sub>2</sub>COCMe: NOH, slightly greenish yellow scales, m. 146-8°, sol. in conc. H<sub>2</sub>SO<sub>4</sub> with orange-red color and gives an intense red-brown color with Ni(OAc)<sub>2</sub> in alc. With NH<sub>2</sub>OH.HCl it forms the dioxime, yellowish, prismatic needles, m. 206-7°, and with PhNHNH<sub>2</sub> the oximephenylhydrazone, cryst. powder, m. 246-8°.

Action of Hydroxylamine on Ketones of the Type RCH: CHCH: CHCOC<sub>6</sub>H<sub>4</sub>. R. CIUSA AND A. TERNI. Ist. chim. gen., univ. Bologna. Atti accad. Lincei, 20, II, 25–30; cf. C. A., 5, 94, 2636.—α-Cinnamylideneacetophenonehydroxylamino-oxime combines with BzH, giving the benzal derivative, C<sub>10</sub>H<sub>20</sub>O<sub>3</sub>N<sub>2</sub>, cryst. powder, m. 175°. Alk. KMnO<sub>4</sub> oxidizes the hydroxylamino-oxime to BzOH and a styrylphenyliso-osasole, PhCH: CHC==CH, gleaming scales, m. 138° (cf. Claus, J. prakt. Chem., 54, 505),

O.N:CPh

while with HNO<sub>2</sub> it gives a compd.,  $C_{17}H_{18}ON$ , isomeric with PhCH: CHCH: CHCPh: NOH and which is the *styryldihydro-osazole*, formed by intramol. addition of the oxime. It m. 124° and forms a *dibromide*,  $C_{17}H_{18}ONBr$ , needles, m. 145°. The hydroxylamino-oxime, boiled with 90% AcOH or dil. mineral acids, gives the  $\alpha$ -oxime. All the above facts are in harmony with the formula PhCH: CHCH(NHOH)CH<sub>2</sub>CPh: NOH for the hydroxylamino-oxime.

New Diethyltrimethylenepyrrole Derivatives. MARIO GHIGLIENO. Atti accad. sci. Torino, 46, 87-95; J. Chem. Soc., 100, I, 321; cf. C. A., 5, 1437-8.—4,4-Diethyl-

trimethylenedicarbonimide-3,5-dicarboxylic acid, HN CO.C(CO<sub>2</sub>H) CEt<sub>2</sub>, needles with

1.5 H<sub>2</sub>O, m. 188-9° (anhydrous acid), is obtained by the action of HNO, on the *monoamide*, m. 247-50°, which, in turn, is prepared from the dinitrile and NaOH. The amide behaves like a dibasic acid, but the NH group is only weakly basic, only about 50% of it being capable of titration with NaOH and phenolphthalein. It forms slightly sol. disilver (with 1 H<sub>2</sub>O), Pb, Cu and Ba salts. The dicarboxylic acid loses 2 CO<sub>2</sub> on heating, forming 4,4-diethyltrimethylenedicarbonimide, yellow, semi-fluid mass giving slightly sol. Ag, Pb, Cu, and Zn salts. The fact that these diethyl derivs. do not exist in isomeric forms like the corresponding 4-methyl-4-ethyl compds. strengthens the theory of spatial isomerism advanced in the case of the latter.

C. A. R.

Relations between Constitution and Phototropy. M. PADOA AND F. GRAZIANI. Lab. chim. gen. r. univ. Bologna. *Gazz. chim. ital.*, 41, I, 385-91; cf. C. A., 4, 2453. Chas. A. ROUILLER.

Relations between α-Benzalphenylhydrazone and some Nitrogen Compounds. R. Ciusa and U. Pestalozza. Ist. chim. gen. r. univ. Bologna. Gazz. chim. ital., 41, I, 391-5; cf. C. A., 4, 1740. Chas. A. Rouiller.

Attempted Asymmetric Synthesis by means of Circularly Polarized Light. M. PADOA. Lab. chim. gen. r. univ. Bologna. Gazz. chim. ital., 41, I, 469-72; cf. C. A., 4, 2452. Chas. A. Rouiller.

Solanine Extracted from Solanum sodomaeum Linn. IV. GIUSEPPE ODDO AND MARCELLO CESARIS. Pavia, ist. chim. gen. univ. Gass. chim. ital., 41, I, 490-534; cf. Ibid., 35, I, 28; 36, I, 310; 36, II, 522.—New analyses of solanine and its salts seem to establish the formula (C<sub>17</sub>H<sub>46</sub>O<sub>2</sub>N)<sub>2</sub>.H<sub>2</sub>O instead of (C<sub>17</sub>H<sub>47</sub>O<sub>2</sub>N)<sub>2</sub>.H<sub>2</sub>O previously given for the base dried in vacuo. It forms 2 series of salts—neutral and basic. Hydrochloride, C<sub>14</sub>H<sub>22</sub>O<sub>12</sub>N<sub>2</sub>.2HCl, pptd. by Et<sub>2</sub>O from abs. alc. Sulfate, turns yellow 150°, decomp. 180-200°. o-Nitrobensoate, minute needles or scales with 8 H<sub>2</sub>O, be-

comes yellow and partially m. 180-200°, m. completely about 205-6° (decomp.), Picrate, horny, amorphous, opaque yellow mass, forming a lemon-yellow powder, evolves gas 110-5°, becomes red 200-22° with partial fusion and decomp. Basic hydrockloride. Basic hydrobromide, prisms or laminas, sometimes clusters of sleuder needles, turns brown about 130°, then decomp. with partial fusion. Basic sulfate, becomes yellow 160-80°, partially fuses 210-5°, decomp. 215-20°. Hydrolyzed with 2% HCl or H<sub>2</sub>SO<sub>4</sub>, the base gives solanidine and a mixture of at least 3 sugars among which d-galactose was identified and a hexose and methylpentose isolated which, while not definitly identified, seem to be glucose and rhamnose. The reducing power of this mixture corresponded to I mol. each of these 3 sugars from I mol. of solanine, and although the amt. of methylpentose, detd. by Votocek's method (Ber., 32, 1195) was more than 1 mol., expt. showed that the method is not applicable to mixtures of sugars, the values ob ained sometimes being high, sometimes low (in the case of galactose it is low, in that of glucose much too high). The mixture showed  $[a]_D^{20}$  25.49° (2.55% soln.). During the hydrolysis the base is reduced by the sugars, as shown by a check expt., to dihydrosolanine, CathatO18N2.H2O, prismatic needles. With NaOAc and Ac<sub>2</sub>O solanine forms a decaacetate, white, floury powder. With AcOH and HNO<sub>2</sub> is obtained a compd. which has a comp. corresponding to the formula C<sub>M</sub>H<sub>m</sub>O<sub>18</sub>N<sub>2</sub>. HNO<sub>2.3</sub>H<sub>2</sub>O, but, besides having 5 ats. of H less than would be required by a solanine nitrite, it does not behave like a salt and is provisionally called azosolanine; it seps. in silky, greenish needles in radiating clusters, almost insol. in H<sub>2</sub>O, sol. in EtOH, turns slightly yellow 230°, evolves gas 240°, blackens and partially m. 275-80°, insol. in dil. mineral acids or in conc. or dil. alk., does not give the nitroso reaction with PhOH-H<sub>2</sub>SO<sub>4</sub> nor the isonitroso test with Ph<sub>2</sub>NH-H<sub>2</sub>SO<sub>4</sub>; conc. KOH ppts. it unchanged from alc. soln. B. in alc. with AcOH or dil. mineral acids it gives an amorphous compd. C<sub>14</sub>H<sub>20</sub>O<sub>21.2</sub>H<sub>2</sub>O, termed oxysolanol, while if the solanine itself, instead of the "azosolanine," is so treated, "solanol," C<sub>54</sub>H<sub>86</sub>O<sub>20-2</sub>H<sub>2</sub>O, needles, turns yellow at 210°, m. partially and decomp. 240-5°, is obtained. CHAS. A. ROUILLER.

Solanidine Sodomaeum. V. GIUSEPPE ODDO. Pavia, ist. chim. gen. r. univ. Gazz. chim. ital., 41, I, 534-52; cf. preceding abstr.—The comp. of soladinine, obtained by hydrolysis of solanine, has been established as being C<sub>18</sub>H<sub>18</sub>ON when dried at 105°, while the product dried in vacuo at room temp. contains 1.5 H<sub>2</sub>O. It does not decolorize Br or KMnO<sub>4</sub> and is therefore not an unsatd. compd. It forms basic and neutral salts. Neutral hydrochloride, C<sub>18</sub>H<sub>18</sub>ON.HCl, elongated prisms, turns yellow at 250° and decomp. 291-2°. Hydrobromide, microscopic prisms, turns markedly yellow at 270°, m. 282-3° (decomp.). o-Nitrobenzoate, prisms, m. 222°. Picrate, lemon-yellow, quadrangular prisms, m. 144-5°. Diacetate, gummy mass yielding the base again on treatment with KOH. With EtMgI the base liberates 2 mols. of C<sub>2</sub>H<sub>4</sub> (cf. C. A., 5, 2809). With AcOH and NaNO<sub>2</sub> the base gives azosolanidine, regular, microscopic prismatic needles with 72.89% C, 9.63% H and 7.94% N. The facts so far known can be represented by the formula HN: C<sub>18</sub>H<sub>29</sub>OH for solanidine, the residue C<sub>18</sub>H<sub>29</sub> being (tri)cyclic.

Bromination of Hydroaromatic Compounds. F. Bodroux and F. Taboury. Compt. rend., 152, 1252-4; cf. C. A., 5, 3232. Chas. A. Rouiller.

Dinaphthothiophene. LANFRY. Compt. rend., 152, 1254-6.—In the action of S on naphthalene at red heat (cf. C. A., 5, 1590) there is obtained in 0.3-0.5% yield a compd. having the comp. of one of the isomeric dinaphthothiophenes (I or II), 1 of which has been described by Henriques (Ber., 27, 2993). It forms light, pearly, yellowish scales, m 250.5° (cor.), b. above 440°, unaffected by cold H<sub>2</sub>SO<sub>4</sub> (66° Bé.), gives no color with isatin in H<sub>2</sub>SO<sub>4</sub> or phenanthroquinone in H<sub>2</sub>SO<sub>4</sub> or AcOH, yields only phthalic acid on oxidation with CrO<sub>2</sub>. With Br it forms a hexabrono derivative,

probably (III) or its isomer, dull yellow, m. 260°, gives 3,6-dibromophthalic acid with CrO<sub>2</sub>. With b. fuming HNO<sub>2</sub>, the thiophene gives a compound C<sub>20</sub>H<sub>2</sub>(NO<sub>2</sub>)<sub>4</sub>S,

yellow powder, m. about 210°, forming yellow solns. with acid reaction, turning red with alk.

Chas. A. Rouller.

Syntheses of α-Ketonic Tertiary Alcohols. D. GAUTHIER. Compt. rend., 152, 1259-61; cf. C. A., 5, 2631.—Blaise's reaction (treatment of aldehyde cyanohydrins with Grignard reagents) is applicable to ketone cyanohydrins, tertiary alc. being formed. 2-Methyl-2-hydroxy-3-butanone, b<sub>730</sub> 79°; semicarbazone, m. 180°. The compd. therefore differs from that to which Schmidt and Austin (Ber., 35, 3724) assigned the same constitution. 2-Methyl-2-hydroxy-3-pentanone, b<sub>730</sub> 96-8°. 3-Methyl-3-hydroxy-2-pentanone, b<sub>730</sub> 94°. Chas. A. Rouiller.

Oximes and Phenylalkylisozazolones Obtained with Ethyl-, Methyl- and Dimethylbenzoylacetic Esters. A. Haller and Ed. Bauer. Compt. rend., 152, 1446-50; cf: C. A., 5, 2093.—The product of the reaction between NH<sub>2</sub>OH.HCl and BzCHEtCO<sub>2</sub>Et is not the oxime HON: CPhCHEtCO<sub>2</sub>H, as assumed by Hantzsch and Miolati (Ber., 26, 1691) but its anhydride, phenylethylisozazolone, m. 91°. Similarly are obtained phenylmethylisozazolone, needles, m. 123-4°, and phenyldimethylisozazolone, m. 70-1°. If the esters are treated with the chlorozincate instead of NH<sub>2</sub>OH.HCl, the oximes are obtained. Ethyl benzoylethyloziminoacetate, prisms. m. 80-1°. Ethyl benzoyldimethyloziminoacetate, needles, m. 135-6°. When hydrolyzed with alc. KOH and acidified, these oximes yield the isoxazolones above.

Catalytic Isomerism of Acetylene Pinacol (Pinacone). Synthesis of Tetramethylketohydrofurane. Georges Dupont. Compt. rend., 152, 1486-8.—HOCMe<sub>2</sub>C: CCMe<sub>2</sub>OH, heated 1 hr. on the H<sub>2</sub>O bath with 0.2 part HgSO<sub>4</sub> and 5 parts H<sub>2</sub>O, is isomerized into tetramethylketohydrofurane, Me<sub>2</sub>C.O.CMe<sub>2</sub>, m. —20.5°, b. 149°, d<sub>18</sub> 0.9251, n<sub>D</sub>

OC-CH.

1.4198, R<sub>M</sub> 38.826. Semicarbazone, needles, m. 190°. Oxime, scales, m. 128°. Phenylhydrazone, m. 134°. With MeMgBr it gives the alc. m. 77° obtained by Bouveault and Locquin by dehydration of the corresponding triol (C. A., 5, 473), while with BrMgC: CMgBr it forms the glycol C<sub>8</sub>H<sub>10</sub>O<sub>2</sub>C: CC<sub>8</sub>H<sub>10</sub>O<sub>2</sub>, m. 97-8°. In certain reactions it behaves like an enol. Thus it gives a sulfate, (C<sub>8</sub>H<sub>13</sub>O<sub>2</sub>SO<sub>4</sub>, hygroscopic, m. 67-70°, and a potassium salt, C<sub>8</sub>H<sub>12</sub>O<sub>2</sub>K, which, with EtI, yields an ethyl ether, b. 157-9°, d<sub>18</sub> 0.8878, n<sub>D</sub> 1.4237, R<sub>M</sub> 48.823. See following abstr.

C. A. R.

Catalytic Preparation of Substituted Ketohydrofuranes. Georges Dupont. Compt. rend., 153, 275-7; cf. preceding abstr.—The transformation of compds. of the type HOCRR/C: CCRR'OH into those of the type RR'C.O.CRR' by means of HgSO<sub>4</sub>

is rapid and quant. when R, R' are aliphatic residues of low mol. wt., but becomes slow and complicated with secondary reactions when they are unsatd., aromatic, or heavier residues. The following compds. have been obtained in this way:  $\alpha, \alpha' - Dimethyl$ 

acetohydrofurane, b. 143°,  $n_D$  I.4267,  $d_{15}$  0.9894,  $R_M$  29.557, reduces Fehling's soln.; semicarbazone, m. 168–70°.  $\alpha,\alpha'$ -Dimethyl- $\alpha,\alpha'$ -diethylketohydrofurane, b. 192°,  $n_D$  I.4368,  $d_{15}$  0.9317,  $R_M$  47.782; semicarbazone, m. 136–8°.  $\alpha,\alpha'$ -Tetraethylketohydrofurane,  $b_{18}$  110°,  $n_D$  I.4486,  $d_M$  0.9275,  $R_M$  57.230.  $\alpha,\alpha'$ -Dipentamethyleneketohydrofurane,  $b_{18}$  152–4°,  $n_D$  I.4904,  $d_M$  I.0268,  $R_M$  62.540.  $\alpha,\alpha'$ -Dissobutylketohydrofurane,  $b_{15}$  112–4°,  $n_D$  I.4385,  $d_M$  0.9066,  $R_M$  57.387. In most cases, during the reaction a ppt. of yellow cryst. scales is formed which, in the case of dimethylketohydrofurane, has the comp.  $C_8H_6(OHg_2SO_4)_6$ . It would seem, therefore, that in the isomerization the OH groups first come into play, the compd. thus formed taking part in the hydration of the triple bond. In the course of the investigation the following compds. were prepared by the Jodsitch method. Diacroleinacetylene, CH<sub>2</sub>: CH(OH)C: CCH(OH): CH<sub>2</sub>  $b_{15}$  146°,  $d_M$  1.0341,  $n_D$  1.5040,  $R_M$  39.515. Dicrotonalacetylene, m. 90–2°; tetrabromide, m. 137°. Dissoraleralacetylene,  $b_{15}$  158–60°,  $n_D$  1.4614,  $d_M$  0.9247,  $R_M$  58.797. Dibutyroneacetylene, pearly scales, m. 120°.

CHAS. A. ROUILLER.

New Method for Obtaining β-Diketones. ÉMILE ANDRÉ. Compt. rend., 152, 1488-90; cf. C. A., 5, 2092.—Diketones are easily obtained by treating compds. of the type R'R'CPh: CHCOR in 80% alc. with acids (e. g., oxalic), pptg. with Cu(OAc), adding a suspension of the Cu salt to dil. HCl and extracting with Et<sub>2</sub>O. C. A. R.

Tetrolic Aldehyde (2-Butinal). P. L. VIGUIER. Compt. rend., 152, 1490-3.— Tetrolic acetal, MeC: CCH(OEt), prepared by Bodroux's method (Compt. rend., 138, 93, 700),  $b_{13}$  60-1°, b. 169-70°,  $d_{16}$  0.902,  $n_D^{16}$  1.4247, when distilled with 10% (CO<sub>2</sub>H)<sub>2</sub>, yields tetrolic aldehyde, b<sub>24</sub> 27-8°, b. 106.5-7.0, needles, m. -26°, d<sub>0</sub> 0.944,  $d_{17}$  0.9265,  $n_{\rm p}^{17}$  1.4467. It rapidly oxidizes to the acid in the air, reduces NH<sub>2</sub>-AgNO<sub>8</sub> and Fehling's soln., colors Schiff's reagent and gives the Fischer-Penzoldt reaction, and is rapidly decomp. by alk. into MeC: CH. Semicarbazone, m. 158°. At low temp. the acetal gives with NH<sub>2</sub>OH.HCl 2-butinal oxime, needles, sublimes about 100°, m. 108-9° and decomp. suddenly a few degrees above the m. p.; it is very unstable, slowly liquefying at room temp. and passing completely, on distillation with steam, into α-methylisoxazole. 2-Butinalhydrazine, from 1 mol. aldehyde and 1 mol.  $N_2H_4$ . $H_2O$ ,  $b_{18}$  63-5°,  $d_{18}$ , 0.9768,  $n^{18}$ . $b_{1}$  1.530; it reacts at once with another mol. aldehyde in the cold to form the azine, yellow needles, m. 123-4°, and with PhNCS to give the phenylthiosemicarbazone, m. 114-6° (decomp.), while if heated with a fragment of solid KOH it gives 5-methylpyrazole. C. A. ROUILLER.

Magnesium Derivative of Fluorene. V. GRIGNARD AND CH. COURTOT. Compt. rend., 152, 1493-5.—Fluorene forms with EtMgBr in PhMe at 135° a Mg deriv. having all the usual properties. Thus, with fluorenone it reacts easily, giving a 75-80% yield of tert.-flourenylfluorenol, m. 195-6°, which, in boiling AcOH, is converted by HCl into the red compd.  $(C_0H_4)_2C$ :  $C(C_0H_4)_2$ , while in EtOH the ether  $(C_0H_4)_2C(OEt)CH-(C_0H_4)_2$ , yellowish needles, m. 168-9°, is obtained, and dry HCl in coid AcOH yields the chloride, slightly yellow, m. 157-8°. With BzPh the Mg compd. gives fluorenyl-diphenylcarbinol, needles, m. 216-7°, which, in EtOH in the presence of HCl, does not form an ether but the compd. Ph<sub>2</sub>C:  $C(C_0H_4)_2$ . Similarly, while  $\alpha$ -indenylfluorenol gives a methyl ether, m. 115-6°, with HCl in boiling MeOH, under the same conditions  $\alpha$ -indenyldiphenylcarbinol is dehydrated.

Chas. A. Rouiller.

Action of Acid Chlorides, Acid Anhydrides and Acetones on Sodium Benzylcyanide. F. Bodroux. Compt. rend., 152, 1594-6; Bull. soc. chim., 9, 726-9.—AcCl and Ac<sub>2</sub>O give poor yields of AcCHPhCN with PhCHNaCN, while BzCl gives 95% of BzCHPhCN. Phthalic anhydride does not react. AcMe and AcPh undergo double decomp.; after treatment with H<sub>2</sub>O, the ketone and PhCH<sub>2</sub>CN are recovered, but with ketones RCOR', where both R and R' are aromatic residues, compds. of the type RR'C: CPhCN

are formed. In this way were obtained Ph<sub>2</sub>C: CPhCN; 1,2-diphenyl-1-p-tolylacrylonitrile, MeC<sub>2</sub>H<sub>4</sub>CPh: CPhCN, needles, m. 123°; and 1,2-diphenyl-1-a-naphthylacrylonitrile, prisms, m. 174-5°. The yields decrease with increasing mol. wts.—from 60% for the first to 8% for the third.

CHAS. A. ROUILLER.

Action of Ammonia on Chloraloses. M. HANRIOT AND A. KLING. Compt. rend., 152, 1596-9; cf. C. A., 5, 3223.—The replacement of Cl in chloraloses by H by the action of NH, in abs. alc. is general. Thus  $\alpha$ -chloralose gives a 25% yield of  $\alpha$ -dechlorochloralose, CaH12OaCl2, needles, m. 165°, [a]15 9.96°; more sol. than the chloralose in H<sub>2</sub>O (4.84% at 15°) and especially in alc. and Et<sub>2</sub>O. Dibenzoyl derivative, C<sub>2</sub>H<sub>10</sub>O<sub>2</sub>-Cl<sub>2</sub>(OBz), obtained by shaking a KOH soln. with BzCl, needles, m. 146°. Decklorochloralic lactone, prepared by heating the chloralose with 5 parts HNO, (d. 1.2), gelatinous mass sol. in NH2 but again sepg. out on conc. Dechlorogalactochloralose, crystals from H<sub>2</sub>O or alc., m. 96°, but when recryst. from CHCl<sub>2</sub> m. 133° and the lower m. form is not again obtained by crystn. from H.O. Et.O or alc. Dibenzoyl derivative, m. 116°. HNO<sub>a</sub> does not give the corresponding acid, but mucic acid, the compd. being apparently hydrolyzed to galactose. Dechlorographinochloralose, m. 88-9°,  $[\alpha]_{D}^{15}$ -19.72°; 15.076 parts dissolve in 100 parts H<sub>2</sub>O at 15°. Dibenzoyl derivative, m. 90.5°. HNO, gives an acid C,H,O,Cl, m. 215°, together with trihydroxyglutaric acid. CHAS. A. ROUILLER.

Ethyl Derivatives of Acetone. Ernst Zerner. Compt. rend., 152, 1599-1601.—By boiling Pr<sub>2</sub>CO with a slight excess of NaNH<sub>2</sub> in C<sub>0</sub>H<sub>6</sub> until no more NH<sub>3</sub> is evolved, then adding 1 mol. Et1, heating 1 hr. longer, fractionating the product and repeating the process several times with the unchanged Pr<sub>2</sub>CO, a practically quant. yield of Et<sub>2</sub>CHCOCH<sub>2</sub>Et, b<sub>741</sub> 174.5-5.5°, is obtained. Et groups can in this manner be successively introduced into the AcMe nucleus, giving (Et<sub>2</sub>CH)<sub>2</sub>CO, b<sub>771</sub> 206-7.5°; pentaethylacetone, b<sub>761.2</sub> 237.5-8.5°; and hexaethylacetone, m. 44°, b<sub>762</sub> 274-5°. The latter is not attacked by an excess of NaNH<sub>2</sub>, either in PhMe or Me<sub>2</sub>C<sub>2</sub>H<sub>4</sub>, even when heated 6 hrs.

Chas. A. Rouiller.

New Method of Esterification of Alcohols by Halogen Acids. G. DARZENS. Compt. 152, 1314-7.—In the presence of tertiary bases (PHNEt<sub>2</sub>, quinoline, pyridine, etc.), SOCl<sub>2</sub> reacts with alc. giving RCl + SO<sub>2</sub> + B.HCl, the yield of ester being 95-100%. The reaction is complete in 20-30 min., no SO<sub>2</sub> being evolved until the product is heated at 80-110°. Practically quant. yields of isoamyl chloride, dichlorohydrin (from chlorohydrin), PhCH<sub>2</sub>Cl<sub>2</sub>Cl<sub>3</sub> PhCH: CHCH<sub>2</sub>Cl, were obtained in this way. Similarly, bromides can be obtained from SOBr<sub>2</sub>. Phenolic OH groups cannot, however, be substituted in this way. See following abstr. C. A. ROULLER.

Action of Thionyl Chloride in the Presence of a Tertiary Base on Esters of Hydroxy Acids. G. Darzens. Compt. rend., 152, 1601-3; cf. preceding abstr.—The formation of chlorides by treatment of an alc. with SOCl<sub>2</sub> in the presence of a tert. base is not limited to alc., but is general for a large no. of compds. containing an alc. OH group. Thus, a 95% yield of MeCHCICO<sub>2</sub>Et is obtained by slowly pouring 125 g. SOCl<sub>2</sub> into 118 g. HOCHMeCO<sub>2</sub>Et in 80 g. pyridine and heating to 110° to expel the SO<sub>2</sub>. PhNMe<sub>2</sub>, PhNEt<sub>2</sub> or quinoline may be used instead of pyridine, altho the product is then somewhat more colored and the yield decreased to 90%. Diethyl chlorosucccinate, b<sub>18</sub> 131°,  $[\alpha]_{20}^{20}$  31° 20′, was made from l-ethyl malate,  $[\alpha]_{20}^{20}$  —11° 58′. In some cases, instead of the chloro esters are obtained ethylene esters, formed by loss of HCl from the former; thus, Et hydroxyhexahydrobenzoate gives Et tetrahydrobenzoate and compounds of the type HOCMeRCH<sub>2</sub>CO<sub>2</sub>Et form the corresponding acrylic esters, CMeR: CHCO<sub>2</sub>Et.

Oxidation of the Higher Acetylenic Acids. A. Arnaud and V. Hasenfratz.

Compt. rend., 152, 1603-6.—Stearolic acid, by the action of alk. KMnO<sub>4</sub>, gives 38% pelargonic and caprylic acids and 55% suberic and azelaic acids, while tariric acid yields 55% lauric and undecylic acids and 25% glutaric and adipic acids. As is known, cautious oxidation of stearolic and tariric acids gives the corresponding diketonic acids, Me(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>),COCO(CH<sub>2</sub>)

2,6-Dibenzoyl-2,6-dimethylheptane and α,α'-Tetramethylpimelic Acid. A. HALLER AND ÉDOUARD BAUER. Compt. rend., 152, 1638-42.—BzCHMe<sub>2</sub>, previously treated in C<sub>0</sub>H<sub>4</sub> on the H<sub>2</sub>O bath with 1 mol. of NaNH<sub>2</sub>, gives, with CH<sub>2</sub>BrCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>Br, a little of a compound b<sub>18</sub> 171-3° (probably BzCMe<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>Br), and 2,6-dibenzoyl-2,6-dimethylheptane, silky needles, m. 248-9°, b<sub>18</sub> 250-5°; dioxime, m. 223-4°. Heated in PhMe with 3 mols. of NaNH<sub>2</sub> the diketone gives the diamide, needles, m. 191-2°, of a,α'-tetramethylpimelic acid, m. 168-9°. With CH<sub>2</sub>ClCH<sub>2</sub>CH<sub>2</sub>Br is obtained 2-benzoyl 2-methyl-5-chloropentane, b<sub>11</sub> 165°, together with (BzCMe<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>. C. A. R<sub>4</sub>

Catalytic Esterification of Aromatic Acids in the Wet Way. J.-B. SENDERENS AND J. ABOULENC. Compt. rend., 152, 1855-7; cf. C. A., 5, 3224.—The same method was used as for formenic acids, except that 2 mols. alc. per mol. acid was taken. With 5 cc. H<sub>2</sub>SO<sub>4</sub>, 61 g. BzOH in 60 cc. 95% alc. gave 41% ester after boiling 3 hrs.; with 10 cc. acid, 75.5% ester was obtained in 40 min.; while with 15 cc. acid the yield of ester was 82% in 20 min. In the presence of substances having but slight affinity for H<sub>2</sub>O (e. g., HKSO<sub>4</sub> or Al<sub>2</sub>(SO<sub>4</sub>)<sub>2</sub>.18H<sub>2</sub>O) there is very little esterification, while the catalytic influence of anhydrous Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> is quite marked. o-Toluic and salicylic acids behave like BzOH. In the case of these acids, therefore, the action of the H<sub>2</sub>SO<sub>4</sub> must depend chiefly on its formation of hydrates. PhCH<sub>2</sub>CO<sub>2</sub>H and PhCH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>H, however, react like aliphatic acids, 2 cc. of H<sub>2</sub>SO<sub>4</sub> with 1 mol. alc. and 0.5 mol. org. acid being sufficient to give 88-92% ester, while KHSO<sub>4</sub> reduces the yield to 76.5% and Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> to 60.5%.

Chas. A. ROUILLER.

Nitration of o-, m- and p-Nitrobenzoyl-p-anisidines. FREDERIC REVERDIN. Compt. rend., 153, 278-9.—o-, m- and p-Nitrobenzoyl-p-anisidines, more or less greenish yellow needles, m. 170°, 174.5° and 197°, resp., when nitrated with HNO<sub>2</sub> (d. 1.52 and 1.4) alone or in AcOH give, according to the conditions, orange 3-nitro-, lemonyellow to pale yellow 2,3-dinitro- and white 2,3,6-trinitro-p- anisidine derivs. A NO<sub>2</sub> group in the Bz residue favors the formation of the higher nitrated anisidines but has no influence on the positions in which the NO<sub>2</sub> groups enter the anisidine mol., but while all the o-nitrobenzoyl derivs. and the trinitro compds. derived from the m- and p-nitrobenzoyl compds. are easily hydrolyzed by heating 1 hr. on the H<sub>2</sub>O bath with conc. H<sub>2</sub>SO<sub>4</sub>, the mono- and dinitro derivs. of m- and p-nitrobenzoyl-p-anisidines are hydrolyzed only with difficulty. (Also in Arch. sci. phys. nat., 32, 124-34.)

CHAS. A. ROUILLER.

Thionic Esters. MARCEL DELÉPINE. Compt. rend., 153, 279-82; cf. C. A., 4, 2934.—The following compds., prepared by the author's original method (Grignard reagents and chlorosulfocarbonic esters) or by that of Matsui (C. A., 3, 2697) are, unless otherwise specified, "oxyluminescent" (oxidize spontaneously with emission of light): Methyl ethanethionate, MeCSOMe, b. 86-9°, d4 1.0062-1.0075. Ethyl ethanethionate, b. 109-10°, d4 0.9816. Ethyl propanethionate, b. 130-2°, d4 0.9639. Methyl isopentanethionate, b. 160-5°, d4 0.9577. Ethyl isopentanethionate, b. 160-5°, d4 0.9549. Methyl isohexanethionate, b. 160-70°(?), non-oxyluminescent. Methyl benzenethionate, b<sub>10</sub> 110-2°. Methyl cyclohexanethionate, b<sub>12</sub> 90-100°, non-oxyluminescent. Methyl nonanethionate, b<sub>13</sub> 115-20°, non-oxyluminescent. Primary and secondary

amines give with these compds. cryst., non-oxyluminescent mono- and dialkylthioamides. Chas. A. Rouiller.

Action of Acids on the Catalytic Oxidation of Phenols by Ferric Salts. H. COLIN AND A. SENECHAL. Compt. rend., 153, 282-3; cf. C. A., 5, 3226.—Expts. on the oxidation of hydroquinone by H<sub>2</sub>O<sub>2</sub> and FeCl<sub>2</sub>, in the presence of AcOH, lactic, malic, tartaric, citric and oxalic acids show that these weak acids have a greater influence than H.SO.. The formation of complex salts can be shown by the disappearance of certain analytical tests for Fe; thus, with 0.04 N H<sub>2</sub>SO<sub>4</sub> and AcOH, KCNS gives a blood-red color; with lactic, malic, tartaric and citric acids, a yellow color; and with (CO<sub>2</sub>H), the greenish color of the ferrioxalate is unchanged. With K<sub>4</sub>Fe(CN)<sub>4</sub> a blue color is formed with all the acids except (CO,H). As the conc. is increased, the differences observed with the different weak acids decrease in intensity, the increase in conc. of the acids increasing the stability of the complex salts. However, the formation of these complex salts is probably not the sole phenomenon concerned, for while the Fe is not detectible by KCNS in the case of a number of acids, the velocity of oxidation is quite different, and many org. substances which form complexes with Fe (sugars, glycerol, AcCH2Ac, etc.) decrease the velocity of oxidation of hydroquinine but slightly. CHAS. A. ROUILLER.

α-Methyllaurenone. New Ketone Derived from Camphor. R. Locquin. Compt. rend., 153, 284-7.—When the lactone  $C_{10}H_{16}O_4$ , m. 189-91°, formed by the action of Caro's reagent on camphor (Baeyer and Villiger, Ber., 32, 3625), is heated 6 hrs. with 16-20 parts 20%  $H_2SO_4$  at 160-5°, in sealed tubes, or with 20%  $H_2PO_4$  at 190-200° it is completely transformed into  $CO_2$  and a ketone  $C_0H_{14}O$ ,  $b_{10}$  82-6°,  $b_{18}$  95-6°,  $d_4^0$  1.062, optically inactive, adds Br in cold CHCl<sub>2</sub> soln. With  $H_2NCONH_2$  it forms 2 semicarbazones, scales, m. 198°, very slightly sol. in b. abs. alc., and another m. 150°. With  $NH_2OH$ , even in excess, it forms a normal oxime,  $C_0H_{14}ON$ , m. 95-6°,  $b_{10}$  122-8° which gives a carbanilideoxime, m.  $101-2^\circ$ . With KMnO<sub>4</sub>, the ketone gives ethyl γ-keto-α,β,β-trimethylvalerate,  $b_{12}$  105-7°; semicarbazone, m. 158-9°. The ketone must, therefore, be 2,3,3,4-tetramethyl-4,5-cyclopentene-1-one or α-methyllaurenone, MeCH. CO.CH. With Na in abs. alc. it yields 2,3,3,4-tetramethyl-1-cyclopentanol,

Me<sub>2</sub>C——CMe

b<sub>10</sub> 87°; phenylurethan, m. 113-4°; CrO<sub>2</sub> oxidizes it to 2,3,3,4-tetramethylcyclopentanone, b. 178-80°, which gives 2 semicarbazones, m. 232° and 182°, resp., the latter being the more sol. in alc.

Chas. A. Rouiller.

Action of Bromine in the Presence of Aluminium Bromide on Cyclohexanol and Cyclohexanone. F. Bodroux and F. Taboury. Compt. rend., 153, 349-50.—Cyclohexanol slowly dropped into a large excess of Br containing 1% of Al gives a small amt. of C<sub>6</sub>Br<sub>6</sub> and a viscous oil which, when the treatment is repeated, yields more C<sub>6</sub>Br<sub>6</sub>. Cyclohexanone similarly treated forms, among other products, a tetrabromocyclohexanone, prisms, m. 117°, decomp. 120-5°. Chas. A. Rouiller.

Action of Anisic and Piperonylic Aldehydes on Sodiated Benzyl Cyanide. F. Bodroux. Compt. rend., 153, 350-1.—Anisic and piperonylic aldehydes give, with PhCHNaCN, 50 and 71%, resp., of MeOC<sub>6</sub>H<sub>4</sub>CH: CPhCN and CH<sub>2</sub>: O<sub>2</sub>: C<sub>6</sub>H<sub>4</sub>CH: CPhCN, together with some of the corresponding acids.

CHAS. A. ROUILLER.

Photolysis of Alcohols, Acid Anhydrides, Ethers and Esters by Ultraviolet Rays. Daniel Berthelot and Henry Gaudechon. Compt. rend., 153, 383-6; cf. C. A., 5, 1400, 1705, 2629.—MeOH and EtOH give H and HCHO and MeCHO, resp. MeAc, after 5 hrs. exposure at a distance of 2 cm. from a Hg lamp (3.6 amp. and 45-50 v.) was decomp. to the extent of 30% with formation of 7 vols. of CO, 13 vols. of H and

80 vols. of CH4. Et,O gave 14.5 vols. of CO and 85.5 vols. of H, CH4 and C,H4, chiefly the latter. Acid anhydrides give less CO, but more CO than the acids; Ac,O yielded 22 vols. of CO<sub>20</sub> 36 vols. of CO, 19 vols. of CH<sub>40</sub>, and 23 vols. of C<sub>2</sub>H<sub>4</sub>. Esters of monobasic aliphatic acids give chiefly CO (more than the alc. or the acid); with CO, it forms about <sup>3</sup>/<sub>3</sub> of the gaseous products, the sum increasing with the wt. of the alc. residue. With a common acid radical, the amt. of CO<sub>2</sub> diminishes with increasing mol. wt., while the CO and the C in the combustible gases increase. With a common alc. residue, the proportion of CO to the combustible gases remains practically 1. AcCH,-CO<sub>2</sub>Et gives 44 vols. of CO, 20.5 vols. of CO<sub>2</sub> and 35.5 vols. of combustible gases yielding 1.46 vols. of CO<sub>2</sub> per vol. of gas. Aromatic esters are much more refractory towards ultraviolet light, PhCH<sub>2</sub>O<sub>2</sub>CH giving 20 times less gas (60% CO and 40% CO<sub>2</sub>) than aliphatic esters and BzOEt and o-HOC, H, CO, Et not being affected at all even when the light was so strong as to heat them above their b. p. Esters of bibasic and hydroxy acids, in general, give chiefly CO (more than 50% in the case of oxalates). The combustible gases from Me esters are rich in CH<sub>4</sub>; those from Et esters in C<sub>2</sub>H<sub>4</sub>. The hydroxy acids give gases particularly rich in CO<sub>2</sub>.

Dithiocamphocarboxylic Acid. L. Chugaev and G. Pigoulevskii. Compt. rend., 153, 388-90.—Camphor, treated in PhMe with NaNH<sub>2</sub>, gives with excess of CS<sub>2</sub> and Me<sub>2</sub>SO<sub>4</sub> methyl dithiocamphocarboxylate, dark yellow liquid, b<sub>2</sub> 179°, d<sup>20</sup> 1.1204,  $[\alpha]_D$  424.58. The acid, a yellow oil, is quite stable at room temp. and is decomp. into camphor and CS<sub>2</sub> on heating.

Chas. A. Rouiller.

Constitution of Divalolactone. S. Losanitch. Compt. rend., 153, 390-2.—Divalolactone gives with MeMgI dimethylanhydrodivalolactone, C<sub>12</sub>H<sub>10</sub>O<sub>2</sub>, b<sub>12-5</sub> 104-5°, [M]<sub>D</sub> 94.85, which, again treated with MeMgI, forms a compound, C<sub>12</sub>H<sub>24</sub>O<sub>2</sub>, b<sub>12-5</sub> 136-7°. α-Methyl-α'-dimethyltetrahydrofurane, b. 102-3°, obtained by dehydration of the corresponding glycol with 60% H<sub>2</sub>SO<sub>4</sub>, does not react with MeMgI. Valerolactone gives 2-methylkexane-2,5-diol, b<sub>14</sub> 121°. Divalolactonic acid and Me<sub>2</sub>SO<sub>4</sub> in the presence of NaOH give, according to the conditions, divalolactone or methyl divalolactonate, b<sub>12</sub> 114°. Divalolactone reacts with PhNHNH<sub>2</sub>, m-O<sub>2</sub>NC<sub>2</sub>H<sub>4</sub>NHNH<sub>3</sub>, NH<sub>2</sub>OH, or H<sub>2</sub>NCONH<sub>3</sub>, giving non-cryst. products. In view of these facts, the author assigns

to it the formula OCHMeCH<sub>2</sub>CHCO instead of that given by Fittig, (Ann., 256, 56;

C,H,CO

267, 191). The structure of the group C<sub>4</sub>H<sub>7</sub> is not yet established. C. A. R.

Technic of the Friedel and Crafts Reaction for Preparing  $\alpha$ -Naphthalenic Ketones without the Formation of the  $\beta$ -Isomers. E. Caille. Compt. rend., 153, 393-4.— Freshly sublimed AlCl<sub>2</sub> is thrown little by little into a CS<sub>2</sub> soln. of naphthalene and acid chloride cooled to  $o^{\circ}$ ; it dissolves and the reaction is accompanied by the pptn. of a cryst. addition product. When the reaction is complete (as detd. by weighing the HCl evolved), the addition product is filtered as rapidly as possible and decomp. with iced HCl. The ketone is obtained in 60-80% yield and so pure that a single crystn. or distillation is sufficient. Oxidation of the products with HNO<sub>3</sub> yields  $\alpha$ -naphthoic acid exclusively. Me, Et, Pr, isobutyl and Ph naphthyl ketones were prepared in this way.

Synthesis of Berberine. Amt PICTET AND A. GAMS. Compt. rend., 153, 386-8; cf. C. A., 5, 2631.—Berberine has been synthesized by condensing homopiperonylamine, CH<sub>2</sub>: O<sub>2</sub>: C<sub>0</sub>H<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>, with homoveratric chloride, (MeO)<sub>2</sub>C<sub>0</sub>H<sub>2</sub>CH<sub>2</sub>COCl, in the presence of NaOH, dehydrating the homoveratrylhomopiperonylamine, needles, m. 136°, thus obtained with P<sub>4</sub>O<sub>10</sub> in boiling xylene, and reducing the resulting compound,

m. 68-70°, with Sn and HCl to veratrylnorhydrohydrostinine, needles, m. 208-10°. This, in conc. HCl on the H<sub>2</sub>O bath, gives with CH<sub>2</sub>(OMe), tetrahydroberberine which is easily oxidized to berberine. See following abstr. Chas. A. ROUILLER.

Stability of the Nitrogen Linkage in the Ketazines. H. WIRLAND AND A. ROSEEU. Chem. Lab., Acad. Sci., Munich. Ann., 381, 229-33.—Benzophenoneketazine, Ph<sub>2</sub>C: NN: CPh<sub>2</sub>, is colorless, but its solns. are yellow, the intensity of the color increasing for different solvents, in the order, alc., Et<sub>2</sub>O, acetone, C<sub>2</sub>H<sub>6</sub> and CHCl<sub>2</sub>. The intensity in C<sub>2</sub>H<sub>6</sub> is about twice that in alc. The azine is not changed by conc. H<sub>2</sub>SO<sub>4</sub>; it gives unstable, additive compds. with dry HCl and Br; the former is lemon-yellow, the latter brilliant orange. Benzalazine, PhCH: NN: CHPh, and benzalbenzophe-

noneazine, Ph<sub>2</sub>C : NN : CHPh, are yellow. Fluorenonehydrazone,  $\begin{vmatrix} C_0H_4 \\ C_2H_4 \end{vmatrix}$  C : NNH<sub>2</sub>

is easily prepared, on the b. H<sub>2</sub>O-bath, from the ketone and N<sub>2</sub>H<sub>4</sub>.H<sub>2</sub>O, in alc.; light yellow plates from alc., m. 149°. Hydrochlorids, deeper yellow. Quinone, or I, in alc., oxidizes the hydrazone to fluorenoneketazine, C, H1, N2; dark red needles with a blue luster from xylene, m. 259°, brick-red when powdered. In conc. H<sub>2</sub>SO<sub>4</sub> the color is purple-red, H<sub>2</sub>O reppts. the azine. The azine does not react with HCl and may be distilled in small quantity. Glacial AcOH and Zn dust reduce the azine to 9-aminofluorene. Tetramethyl-p-diaminobenzophenonehydrazone, (Me,NC,H4),C: NNH2, prepared at 180°, in the manner described above; light yellow needles from alc., m. 150°. It resembles auramine in its behavior towards acids. In glacial AcOH the color is orange-red. Benzylidene derivative, CyHzeN4, from the hydrazone and BzH, in alc.; light orange-yellow needles from alc., m. 141°. It is insol. in dil. AcOH, but gives an orange-red color with dil. mineral acids. Tetramethyl-p-diaminobenzophenoneketazine, (Me,NC,H,),C: NN: C(C,H,NMe,), from the hydrazone and HgO, in presence of a little I and xylene; large, brownish red crystals from xylene, m. 253°. When powdered the color is orange-yellow; in dil. acids light red, in glacial AcOH, brilliant red, in conc. H,SO, colorless. The stability of this ketazine is essentially similar to that of the others described above. J. BISHOP TINGLE.

Constitution of Auramine. LEOPOLD SEMPER. Chem. Lab., Roy. Acad. Sci., Ann., 381, 234-64.—The auramine base is obtained colorless by treating the purified hydrochloride ("auramine") with dil., aq. NaOH and C<sub>2</sub>H<sub>4</sub>, at the ordinary temp., the C<sub>e</sub>H<sub>e</sub> being subsequently evapd. under reduced pressure, in an atm. of H or N. Aggregates of lustrous, white crystals which are stable to light. When the base is warmed with dil. alc. and phenolphthalein an orange-red color is produced; it exhibits the characteristic absorption bands of the quinoidal phenolphthalein salts and its production indicates that a portion of the auramine base has combined with H<sub>2</sub>O to form an ionium hydroxide. Impure auramine carbonate is formed by passing CO. into a moist, ethereal soln. of the base, or from moist CO, and the powdered base. The carbonate is not formed in the absence of H<sub>2</sub>O. The soln. obtained by passing CO, thru an aq. suspension of the base gives a well-marked alk., reaction with curcuma paper, provided that all the base is not in soln. A satd., aq. soln. of the hydrochloride gives only a slight color with curcuma paper. Auramine perchlorate, from the base and HClO4, in Et2O or C2H4; yellow crystals. The base gives an orange-yellow color with PhOH, changing to yellow with alc., or Et<sub>2</sub>O, and it forms additive compounds with HgCl<sub>2</sub> (egg-yellow) and SnCl<sub>4</sub> (light orange). The hydrochloride also gives an additive compound with AgNO<sub>2</sub>; yellow needles. When b. with alc., AgCl is pptd. Dry, or ethereal HCl, in excess, or glacial AcOH produces a deep orange-color with the base, it is due to the formation of an unstable dihydrochloride. Similar effects are produced by other acids; the colored materials are probably mixtures of polyacidic salts. A det. of the elec. cond., at 25°, gave the following results: auramine hydrochloride ("auramine") μ<sub>442</sub> 87.40; after adding NaOH and correcting for NaCl, μ<sub>1024</sub>, 23.7 after 140 min. without stirring. When the liquid was stirred the same (constant) value was obtained after 14 min. Hantzsch and Oswald obtained the initial values 25.6 and 47.7, at 0° and 25°, resp. (Ber., 33, 283 (1900)). If the change which takes place on adding NaOH to auramine hydrochloride includes a rearrangement to a quinoidal system, its velocity should be lower at 0° than at 25° and no effect should be produced by stirring. Moreover, the initial cond., which decreases with time and therefore with the progress of this assumed change, should be higher at 0° than at 25°. The results show the exact opposit of this and demonstrate that the auramine base forms a supersatd. soln., the changing cond. simply measures the velocity of the cryst. of the

auramine base. Acetylauramine, (Me,NC,H<sub>4</sub>),C: NAc, is prepared by treating the base with Ac<sub>2</sub>O, at room temp., the product being treated with alc. and then with dil., aq. Na<sub>2</sub>CO<sub>2</sub>; slender, pale yellow needles, m. 221°. It quickly turns blue on exposure to acid vapor, or to CO<sub>2</sub> of the air. Quinoidal hydrochloride, Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>C(NHAc): C<sub>6</sub>H<sub>4</sub>: NMe.Cl.EtOH, from the preceding compd. and alc. HCl, at o°. Et.O being then added; heavy needles with a green luster. The color is violet-blue in H<sub>2</sub>O, in thick layers red; in glacial AcOH, or alc., bluish violet, in acetone, reddish violet and in CHCl,, violetred. The alc. soln. exhibits well-defined absorption bands with a maximum at  $\lambda$  585 and also absorbs in the blue and violet regions (see below). In H<sub>2</sub>O the hydrochloride decomp. spontaneously into tetramethyl-p,p'-diaminobenzophenone and AcNH<sub>2</sub>. At oo, HCl, in Et,O, gives a highly unstable dihydrochloride, orange-flocks. Acetylauramine also forms deeply colored compounds with certain metallic halides, such as SnCl. Mercuric chloride, crystallin. Methiodide, Me, NC, H, C(NMeAc): C, H, : NMe.I. is prepared in dry CHCl<sub>2</sub>; unstable, crystallin and hygroscopic. It resembles the above quinoidal hydrochloride in its reactions and in the character of its absorption, but the maximum is further in the red,  $\lambda$  600. With alkali in excess, it gives a skyblue color. Ethiodide and dimethyl sulfate, resemble the preceding compd., but are less stable. Picryl chloride derivative, reddish violet and unstable. Acetylauramine is recommended for use as an indicator for acids; they change its color from yellow to Its dissociation constant is 10<sup>-7</sup> to 10<sup>-8</sup>, that of phenolphthalein being 10<sup>-7</sup>. By means of the Ac compd. it is easy to show the presence of CO<sub>2</sub> in ordinary H<sub>2</sub>O. It is best employed in connection with dil. solns., which may be alc. and it is especially applicable to slightly colored liquids, particularly those of a yellow shade. Benzoylauramine hydrochloride, Me<sub>2</sub>NC<sub>2</sub>H<sub>4</sub>C(NHBz): C<sub>2</sub>H<sub>4</sub>: NMe<sub>2</sub>Cl, is prepared at o° from HCl, in Et.O. It resembles the Ac compd., but is less stable and crystallizes with greater difficulty. Its maximum of absorption is at  $\lambda$  590. Phenylsuljonylawamine, (Me,NC,H,e),C: NSO,Ph, from auramine and phenylsulfonyl chloride, in C.H., at the ordinary temp.; yellow, lustrous prisms from AcOEt, m. 182. In PhOH, or glacial AcOH, the color is reddish brown. Hydrochloride, deep red with a violet tinge, very unstable. Its spectrum resembles that of the Ac salt, the maximum being at about  $\lambda$  570. The sulfonyl deriv. forms unstable compounds with oxalic acid, in Et<sub>2</sub>O, and also with SnCl.. The former is dull green, changing to yellow, the latter is green and flocculent. 4-Nitrophenylauramine, (Me<sub>2</sub>NC<sub>2</sub>H<sub>4</sub>)<sub>2</sub>C: NC<sub>2</sub>H<sub>4</sub>NO<sub>2</sub>, is prepared from the auramine base and p'-nitroaniline, at 160°; dark yellow crystals from amyl alc., or AcOEt, m. 226°. Yield, 80% of the auramine. In ordinary solvents the color is vellow to orange, in PhOH, or glacial AcOH, blood-red. Hydrochloride, from HCl, in and flocculent. 2,4-Dinitrophenylauramine, (Me,CC,H,),C: blood-red NC<sub>2</sub>H<sub>2</sub>(NO<sub>2</sub>)<sub>2</sub>, from 2,4-dinitrochlorobenzene and the base (2 mol.), in C<sub>2</sub>H<sub>2</sub>, during 3 dys.; brick-red powder from amyl alc., m. 198°. Its solns, are orange; the color with PhOH, or glacial AcOH is dark red. Hydrochloride, dark red, hygroscopic flocks. 2,4,6-Trinitrophenylauramine, (Me2NCaH4)2C: NCaH2(NO2)2, is prepared like the dinitro compd., from picryl chloride; lustrous, red needles from amyl alc., m. 211%. From CaHa large, lustrous prisms with 0.5 CaHa are deposited. Its solns. are orangered; it is decomp. rather slowly by b., dil., alc. HCl. Hydrochloride, dark reddish brown flocks, which soon change to needles with a bronze luster. It is fairly stable and its solns, are all brownish red and of a lighter shade than the above dinitrophenyl hydrochloride. The absorption spectra of auramine base, of its hydrochloride and of the above mono-, di- and trinitrophenyl derivs., in alc., are similar, differing only in the slightly varied position of the max. of absorption. Consequently they all have the benzenoidal, imine formulas given above. The acetylauramine salts have a different spectrum, similar to that of tetramethyldiaminobenzhydrol and are undoubtedly quinoidal. The com. dye "auramine" is the hydrochloride. (Me,NC,H,),C: NH,HCl, of the colorless, secondary amine (Me2NCeH4)2C: NH, which, chemically, is termed auramine, or tetramethyl-p,p'-diaminodiphenylamine. Tetramethyl-p,p'-diaminobenzophenone gives an intensely yellow color in PhOH, or glacial AcOH. In anhydrous Et<sub>2</sub>O, at o°, (CO<sub>2</sub>H)<sub>2</sub> gives an orange-colored, unstable, flocculent salt. Stannic chloride, C12HanON2. SnCl4, is formed at oo, in CaH4 + gasolin; scarlet-red and flocculent; it is instantly decomp. by atm. H<sub>a</sub>O. Perchlorate, orange-colored and oily.

J. BISHOP TINGLE.

Oxyazo. Compounds and Ketohydrazones. IV. Phenylhydrazone. Derivatives of 4-Methylketocoumaranone. K. Auwers and R. Apitz. Chem. Inst., Univ. Greifswald. Ann., 381, 265-76; see C. A., 5, 877.—4-Methylketocoumaranone-β-phenylhydrazone formula (I) below, is prepared from the ketone and PhN<sub>2</sub>H<sub>2</sub>.HCl, in hot, glacial AcOH; bright yellow needles from dil. AcOH, m. 148°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is

$$MeC_eH_s$$
 $C(N:NNHPh)$ 
 $CO$ 
 $MeC_eH_s$ 
 $C(:NNPhBz)$ 
 $CO$ 
 $(II)$ 

brownish red. Dil., alc. NaOH hydrolyzes it, at the ordinary temp., to 4-methyl-rhydroxyphenylglyoxalic phenylhydrazone, HOCaHaMeC(: NNHPh)COaH, which was also prepared from 4-me.hyl-1-hydroxyphenylglyoxalic acid and PhN<sub>2</sub>H<sub>2</sub>, in alc.; greenish yellow needles from dil. alc., m. 160° (decomp.). It regenerates (I) when warmed with C<sub>6</sub>H<sub>6</sub> and P<sub>4</sub>O<sub>10</sub>, or when b. with glacial AcOH. 4-Methylketocoumaranone-βbenzoylphenylhydrazone (II), is prepared in dil. alc., at the ordinary temp., from βbenzoylphenylhydrazine; white needles from MeOH, m. 168-9°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is orange-yellow. In AcOEt, at the ordinary temp., Zn dust and glacial AcOH reduce it to benzanilide. It is hydrolyzed by dil., alc. NaOH, at the ordinary temp., to BzOH and the above 4-methyl-1-hydroxyphenylglyoxalic phenylhydrazone, to a mixture of these and 4-methyl-1-hydroxyphenylglyoxalic acid \( \beta \)-benzoylphenylhydrazone, HOC, H, MeC(: NNPhBz)CO, H, or to this last compd. only, the nature of the product depending on the duration of the hydrolysis; small, white crystals from MeOH, m. 112° (decomp.). In conc. H.SO, the color is orange-yellow. B., aq. NaOH quickly hydrolyzes it to the above phenylhydrazone (m. 160°). 4-Methyl-1-hydroxyphenylglyoxylic phenylhydrazidephenylhydrazone, HOC, H, MeC(: NNHPh)CONHNHPh, is prepared by b. 4-methylketocoumaranone with alc. and PhN<sub>2</sub>H<sub>2</sub>; white needles, m. 183°. It reduces hot Fehling's soln. and the resulting liquid, when acidified, deposits (I). In hot, aq. NaOH the color is yellow, in conc.  $H_rSO_4$ , orange-yellow. 4-Methylketocoumaranone- $\alpha$ -phenylhydrazone (III), is prepared from 4-methylketo-

$$MeC_6H_3 < CO > C : NNHPh$$
(III)

coumaranone and phenyldiazonium chloride, in aq. alkaline, or AcOH soln.; lustrous, thin, golden yellow plates from alc., or acetone, m. 224°. The color in conc. H<sub>4</sub>SO<sub>4</sub> is cherry-red. Under similar conditions (III) is formed 4-methyl-1-aceto- (or benzo-)-2-hydroxycoumarone and phenyldiazonium chloride. Attempts to acetylate, or to benzoylate (III) failed. 4-Methylketocoumaranoncosazone (IV), is prepared by b.

$$MeC_{e}H_{s}$$
 $C(:NNHPh)$ 
 $C:NNHPh$ 
 $O$ 
 $(IV)$ 

 $PhN_2H_3$  and (III), or at 100°; golden yellow plates from glacial AcOH, m. 223° (decomp.). In conc.  $H_2SO_4$  the color is brownish violet. The compd. does not reduce b. Fehling's soln. The reactions described above establish the formulas given and show that the compds in question are true phenylhydrazones.

J. B. T.

Double Salts of Antimony Pentachloride and Alkaloid Hydrochlorides. Th. Sv. Thomshn. Oversigt o. d. Kgl. Danske Vidensk. Selskabs. Forhandl., 1911, 41-55; thru Chem. Zentr., 1911, I, 1515.—The compds. described below were prepared by dissolving the resp. alkaloids in alc. (90%), adding HCl (40%) and then SbCl<sub>8</sub>, in HCl (40%). Quinine, C<sub>20</sub>H<sub>21</sub>O<sub>2</sub>N<sub>2.2</sub>HCl.SbCl<sub>8</sub>, heavy, S-yellow, cryst. powder-Quinidine, C<sub>20</sub>H<sub>21</sub>O<sub>2</sub>N<sub>2.2</sub>HCl.SbCl<sub>6</sub>, yellow, microscopic, triangular prisms. C n-chonine, C<sub>10</sub>H<sub>21</sub>ON<sub>2.2</sub>HCl.SbCl<sub>8.2</sub>H<sub>2</sub>O, light yellow, microscopic, prismatic rods. Cin. chonidine, C<sub>10</sub>H<sub>21</sub>ON<sub>2.2</sub>HCl.SbCl<sub>8.2</sub>H<sub>2</sub>O, light yellow, rectangular, microscopic plates. Morphine, 2C<sub>17</sub>H<sub>19</sub>O<sub>4</sub>N.2HCl.SbCl<sub>8.4</sub>H<sub>2</sub>O, large, light brown plates. Codeine, 2C<sub>18</sub>H<sub>21</sub>O<sub>4</sub>N.2HCl.SbCl<sub>8</sub>, reddish brown, microscopic plates. Strychnine, C<sub>21</sub>H<sub>22</sub>O<sub>3</sub>N<sub>3.4</sub>HCl.SbCl<sub>8</sub>, heavy, red, rhombic plates and short, rhombic prisms. Cocaine, C<sub>17</sub>H<sub>21</sub>O<sub>4</sub>N.HCl.SbCl<sub>8</sub>, colorless, lustrous scales, or thin, irregular, microscopic platelets. Caffeine, C<sub>4</sub>H<sub>10</sub>O<sub>4</sub>N<sub>4</sub>.HCl.SbCl<sub>8</sub>.H<sub>2</sub>O, yellow, crystals. Nicotine, 2C<sub>10</sub>H<sub>14</sub>N<sub>2.4</sub>HCl.3SbCl<sub>8.3</sub>H<sub>4</sub>O, pale rose-red needles, or microscopic, cylindrical rods. J. B. T.

Hypaphorine and its Relationship to Tryptophan. P. VAN ROMBURGH. Org. Chem. Lab., Univ. Utrecht. Kon. Akad. v. Wetensch. Amsterdam, Wisk. en. Natk. Afd., 19, 1250-3; thru Chem. Zentr., 1911, I, 1548-9.—Hypaphorine, C<sub>14</sub>H<sub>18</sub>O<sub>2</sub>N<sub>2</sub>, is an alkaloid which occurs in the seeds of Erythrina hypaphorus Boerl; crystals, m. 255° (decomp.); [a]<sub>0</sub> 91-3°. When heated strongly it burns and evolves vapors having an indole odor. Hydrochloride, C<sub>14</sub>H<sub>18</sub>O<sub>2</sub>N<sub>2</sub>,HCl. Hot, aq. KOH hydrolyzes hy-

possible formula for hypaphorine, in which case its relationship to tryptophan is obvious.

J. BISHOP TINGLE.

Colchicine. I. A. WINDAUS. Sitzungsb. I. Heidelberger Akad. I. Wiss., 1910, 1-7; thru Chem. Zentr., 1911, I, 1637-8.—In addition to the compds. described previously (C. A., 4, 2665), the author has obtained 3,5,6-trimethoxyphthalic acid, (MeO), CeH(CO,H), as a product of the oxidation of colchicine, by means of aq. KMnO, and KOH (5%). It is best purified thru the anhydride; transparent plates from H<sub>2</sub>O, m. 175-6° when quickly heated. At a higher temp. it gives a sublimate of the

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anhydride. This latter is obtained by heating the crude oxidation product at 220°, under reduced pressure. Barium salt, needles from dil. alc. Anil, C<sub>17</sub>H<sub>14</sub>O<sub>4</sub>N, color-less needles from dil. alc., m. 146° (see following abstr.).

J. BISHOP TINGLE.

Colchicine. II. A. WINDAUS. Med. Dept. Lab., Univ. Freiburg i/B. Situngsber. I. Heidelberger Akad. I. Wiss., 1911, 1-27; thru Chem. Zentr., 1911, I, 1638-41; see preceding abstr.—Zeisel has stated the colchiceine, which is formed by the interaction of H<sub>2</sub>O and colchicine, contains a CO<sub>2</sub>H group, colchicine being its Me ester. The author regards the compds. as enol and Me ether, resp. In contrast to colchicine, colchiceine gives a green color with FeCla. The relationship of colchicine and its more important derivs. may, at present, be represented as follows: colchicine, (MeO)<sub>2</sub>C<sub>a</sub>H : C<sub>10</sub>H<sub>2</sub>O(OMe)NHAc; colchiceine, (MeO)<sub>2</sub>C<sub>6</sub>H : C<sub>10</sub>H<sub>2</sub>O(OH)NHAc; trimethylcolchicinic acid, (MeO)<sub>2</sub>C<sub>6</sub>H: C<sub>10</sub>H<sub>2</sub>O(OH)NH<sub>2</sub>. The characteristic, yellow color, produced by adding HCl to colchicine, or to its derivs., is due to the formation of additive compds., possibly oxonium salts. Trimethylchicinic acid dihydrochloride, C<sub>10</sub>H<sub>21</sub>O<sub>4</sub>N.2 HCl.H<sub>2</sub>O, from the hydrochloride and excess of HCl, at a low temp.; dark yellow crystals. At 100° it regenerates the monohydrochloride and it contains enolic OH because it reacts with FeCl, like the monohydrochloride. Trimethylcolchicinic acid and its hydrochloride are best purified thru the monohydrochloride. Dibensoyltrimethylcolchicinic acid, (MeO), C.H: C, H.O(OBz)NHBz, from the hydrochloride and BzCl, in pyridine; pale yellow, triangular prisms from acetone, m. 298° (decomp.). It gives no color with FeCl,. N-Benzoyltrimethylcolchicinic acid, (MeO), CaH: C10HaO(OH)NHBz, from the preceding compd. and alc. KOH, at 100°; pale yellow needles from alc. (95%), m. 253-4°. Its color with FeCl, is dark green and it does not affect blue litmus paper. N-p-Nitrobenzoyl derivative, prisms, m. 256°. N-p-Bromo-N-Phenylsulfonyl derivative, formed by means of aq. KOH benzoyl derivative. (10%); greenish yellow, rhombic plates from alc., m. 255°. Its color with FeCl, is intensely green. Bisphenylsulfonyl derivative, C12H12O4(OSO2Ph)NHSO2Ph, prepared in pyridine; quadratic, rhombic plates from 95% alc., m. 196°. The mother liquor contains an isomeric compound; greenish yellow prisms and plates from H<sub>2</sub>O, m. 141-2°. The compds. are probably cis- trans- isomers, because neither gives a color with FeCla and each is hydrolyzed to the same monophenylsulphonyl compd. At oo, in presence of KOH, aq. KMnO<sub>4</sub> (4%) oxidizes N-benzoyltrimethylcolchicinic acid to N-benzoylcolchid formula (I) below; needles from alc., m. 239°; in CHCl<sub>a</sub>,  $[a]_D^{17}$  —275°. It does not react with litmus paper, nor with FeCl, and it gives when, oxidized, 3,4,6-trimethoxyphthalic acid. At 250°, under reduced pressure (I), gives benzamide and

trimethoxyhomonaphthid (II); needles from H<sub>2</sub>O, m. 138°. It is also formed, together with a little colchid, from (I) and alc. HCl. N-Bensoylcolchinic anhydride (III), from

N-benzoyltrimethylcolchinic acid, aq. KOH and KMnO<sub>4</sub>; yellow prisms, or long, quadratic plates from C<sub>4</sub>H<sub>6</sub>, m. 207°; in CHCl<sub>2</sub> [ $\alpha$ ]<sup>18</sup><sub>D</sub> —288. The solns. in neutral media are intensely yellow, in alkalies colorless. Anil, C<sub>2</sub>H<sub>20</sub>O<sub>4</sub>N<sub>2</sub>, almost colorless needles from dil. alc., m. 226°. At 100°, AcOH (75%) and Zn dust convert (III) into a compound (IV); white needles from dil. acetone, m. 158°, previously softening. It is very stable towards KMnO<sub>4</sub>. Warm HI (d. 1.72) and AcOH hydrolyze both (III) and (IV) to the compound (V); colorless needles from 75% AcOH, darkens above

200°, decomp. about 230°. It is neutral to litmus. N-Acetylcolchid vide (I), is prepared from colchicine and KMnO<sub>4</sub>; needles from MeOH, m. 221°. N-Acetylcolchinic anhydride is formed in small quantity, together with the preceding compd.; deep yellow needles, m. 201°. In alkalies its solns. are colorless. N-Phenylsulfonylcolchinic anhydride,  $C_{12}H_{11}O_{2}NS$  (vide III), is prepared at 0°, from N-phenylsulfonyltrimethylcolchicinic acid, aq. KOH and KMnO<sub>4</sub>; long, quadratic plates from CHCl<sub>4</sub> + petroleum ether, dil. acetone, or dil. AcOH, m. 242-3° (decomp.). In AcOH the color is deep yellow; it is discharged quickly by adding Zn dust. In dil. aq., KOH no color is produced. The fact that (III) and the N-phenylsulfonyl anhydride are both acyl derivs. of the same compd. has been demonstrated by hydrolyzing each substance and converting the resulting product into its complimentary acyl deriv. The formulas given above are discussed; they are largely provisional.

J. B. T.

p-Nitrosomethylethylaniline, a New Intermediate Product for the Dye Industry. JOHN CANNELL CAIN. Rep. 7th Intern. Congress, London, Sec. IV, B., 1909; thru Chem. Zentr., 1911, I, 1742.—p-Nitrosomethylethylaniline, ONC<sub>2</sub>H<sub>4</sub>NMeEt, is prepared from PhNMeEt, HCl and HNO<sub>2</sub>; green plates from dil. alc., m. 66-7°. Hydrochloride, yellow needles. The base gives a dye of the methylene blue series; it is termed "methylene blue M. E" (M. E = methyl ethyl), it differs from methylene blue in having Et in place of Me. It dyes cotton, mordanted with tannin, a more intense green than is produced by methylene blue itself.

J. BISHOP TINGLE.

Orthophosphoric Acid as a Dehydrating Catalytic Agent. I. Condensation of Acetone in Presence of Phosphoric Acid. PANCHANAN NEOGI. Chem. Lab., Rajshahi Col., Eastern Bengal, India. J. Chem. Soc., 99, 1249-52; Proc. Chem. Soc., 27, 7.—Acetone (50 cc.) and H<sub>2</sub>PO<sub>4</sub> (d. 1.75, 40 cc.) when mixed, cooled and allowed to remain over night, undergo but little change, but a brown liquid is obtained if the acetone be mixed with H<sub>2</sub>PO<sub>4</sub> (2 pts.) and action allowed to proceed during 2 dys. The volatil products consist of mesityl oxide, mesitylene, phorone and xylitone, together with varying quantities of unchanged acetone. The original H<sub>2</sub>PO<sub>4</sub>, after the distillation, is found to be converted completely into H<sub>4</sub>P<sub>2</sub>O<sub>7</sub> and HPO<sub>3</sub>. This mixture of acids is capable of causing the condensation of at least 4 more portions of acetone (70-50 cc. each). The use of H<sub>2</sub>PO<sub>4</sub> offers distinct advantages over that of H<sub>2</sub>SO<sub>4</sub>, or HCl, the former causes charring and the latter volatilizes during the distillation. No organic intermediate products of P could be isolated.

J. BISHOP TINGLE.

Trialkylammonium Nitrites and Nitrites of the Bases of the Pyridine and Quinoline Series. I. Panchanan Neogi. Chem. Lab., Rajshahi Col., Eastern Bengal, India.

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J. Chem. Soc., 99, 1252-4; Proc. Chem. Soc., 27, 71.—Triethylammonium nitrite, Et<sub>2</sub>N.HNO<sub>2</sub>.H<sub>2</sub>O, is prepared by grinding Et<sub>2</sub>N.HCl and AgNO<sub>2</sub> with a little H<sub>2</sub>O, the mixture is well shaken and solns. of the chloride and AgNO<sub>2</sub> are added until neither of them gives a ppt. with a drop of the filtered mixture. Under reduced pressure, at the ordinary temp., the clear liquid deposits very hygroscopic, colorless crystals; decomp. violently when heated in an open tube, m. about 60° (10-20 mm.) sublimes 70-5°, a part decomp. The salt is highly hygroscopic and is volatil with steam, especially under reduced pressure. Yield, almost quant. At the ordinary temp., the nitrite passes slowly into the nitrate. Pyridinium nitrite was prepared at 0°, in the manner described above. Like Et<sub>2</sub>N.HNO<sub>2</sub>, its soln. liberates I from KI + HCl, but the only crystals which could be obtained consisted of pyridinium nitrate. In spite of the title of the paper, no expts. are described with other amines.

J. BISHOP TINGLE.

Aromatic Antimony Compounds. II. Action of the Chlorides of Antimony on Aniline and its Derivatives. PERCY MAY. Univ. College, London. J. Chem. Soc., 99, 1382-6; Proc. Chem. Soc., 27, 124; see C. A., 5, 480.—The following compds. or SbCl, were prepared from the resp. amines mentioned below. Trianiline antimony trickloride, (PhNH<sub>2</sub>),SbCl<sub>2</sub>, by adding PhNH<sub>2</sub> to the chloride, in C<sub>2</sub>H<sub>2</sub>; needles from alc., m. 130-70° (decomp.). It is hydrolyzed by H<sub>2</sub>O, or alkali and, with alc., forms PhNH, HCl. At 210°, PhNH, and SbCl, give trianiline antimony trichloride dihydrochloride, (PhNH<sub>2</sub>)<sub>2</sub>.SbCl<sub>2</sub>.2HCl; colorless crystals from alc., m. 170-5° (decomp.). o-Toluidine antimony trichloride, MeC, H, NH, SbCl, was prepared in C, H, long, colorless needles from alc., m. 148°. p-Toluidine antimony trichloride, prepared like the isomer; colorless crystals, decomp. above 120°. p-Chloroaniline antimony trichloride, ClC<sub>e</sub>H<sub>4</sub>NH<sub>2</sub>.SbCl<sub>2</sub>, obtained from C<sub>e</sub>H<sub>6</sub> soln.; white, insol. powder, m. 160-5° (decomp.). SbCl, does not combine with m- or p-nitroaniline. Charring takes place when SbCl, is added to CaH, toluene, xylene, or to PhNH, in CHCl. Unstable crystals are deposited from a soln. of SbCl, in CHCl, J. BISHOP TINGLE.

Synthesis of Histidine. Frank Lee Pyman. Wellcome Chemical Works, Dartford, Kent. J. Chem. Soc., 99, 1386-401; Proc. Chem. Soc., 27, 92.—Ethyl 4(or 5)-glyoxalinemethylmalonate formula (I) below, was prepared from 4(or 5)-chloromethylglyoxaline hydrochloride and Et sodiomalonate (2 mols.); yellow, viscid oil. Yield, 49%. Hydrochloride, deliquescent needles, m. 50-70° (cor.). Acid oxalate, C<sub>11</sub>H<sub>16</sub>O<sub>4</sub>N<sub>2</sub>.C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>, large, hard, clear, nearly rectangular, oblong plates from H<sub>2</sub>O, m. 155-8° (decomp.). Hydrodide, crystallin. Hydrolysis of (I) by means of b.

aq. Ba(OH)<sub>2</sub> gives 4(or 5)-glyoxalinemethylmalonic acid; clear, hexagonal plates from H<sub>2</sub>O, m. and evolves CO<sub>2</sub> 180° (cor.), becomes solid, then m. 207° (cor.). The compd. formed by the elimination of CO<sub>2</sub> is  $\beta$ -glyoxaline-4(or 5)-propionic acid; it is also present in the mother liquor from the preceding malonic acid deriv. Ethyl 4(or 5)-glyoxalinemethylacetoacetate (II), is prepared from 4(or 5)-chloromethylglyoxaline hydrochloride and Et sodioacetoacetate (2 mols.). Acid oxalate, C<sub>10</sub>H<sub>14</sub>O<sub>2</sub>N<sub>2</sub>.C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>, aggregates of thin, clear plates from H<sub>2</sub>O, m. 145-6° (cor.) (decomp.). Ethyl 4(or 5)-glyoxalinemethylmethylacetoacetate, C<sub>2</sub>H<sub>3</sub>N<sub>2</sub>CH<sub>2</sub>CMe(COMe)CO<sub>2</sub>Et, is obtained in a similar manner to (II) from Et sodiomethylacetoacetate. Hydrochloride, deliquescent plates. Acid oxalate, (C<sub>11</sub>H<sub>16</sub>O<sub>3</sub>N<sub>2</sub>)<sub>4</sub>·3C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>, clear, lustrous plates from H<sub>2</sub>O, m. 155-6° (cor.) (decomp.). Ethyl 4(or 5)-glyoxalinemethylchloromalonate, C<sub>2</sub>H<sub>2</sub>N<sub>2</sub>CH<sub>2</sub>CCl(CO<sub>2</sub>Et)<sub>2</sub>, is formed by adding 4(or 5)-chloromethylglyoxaline hydrochloride to a mixture of

Et chloromalonate and abs. alc.; viscid oil. Hydrochloride, C<sub>11</sub>H<sub>12</sub>O<sub>4</sub>N<sub>3</sub>Cl.HCl, large, glistening, diamond-shaped plates from acetone, m. 148–9° (cor.). It gives ppts. with picric acid, or Meyer's solns. and a deep red color with alkaline Na diazobenzene-p-sulfonate. Acid oxalate, (C<sub>11</sub>H<sub>15</sub>O<sub>4</sub>N<sub>3</sub>Cl)<sub>4·3</sub>C<sub>5</sub>H<sub>2</sub>O<sub>4</sub>, shimmering leaflets from H<sub>2</sub>O, m. 176° (cor.) (decomp.). Hydrolysis with HCl (20%) converts the base into dl-α-chloro-β-glyoxaline-4(or 5)-propionic acid, C<sub>2</sub>H<sub>2</sub>N<sub>3</sub>CH<sub>2</sub>CH<sub>2</sub>CHClCO<sub>2</sub>H; stellate clusters of white, prismatic needles from H<sub>2</sub>O, m. 201° (cor.) (decomp.). Yield, 91%. The acid described in the literature (m. 191°) is probably the isomer corresponding with l-histidine (see below). With b., dil., aq. NaOH, Et 4(or 5)-glyoxaline-methylchloromalonate evolves NH<sub>2</sub> (f mol.); aq. NH<sub>3</sub>, at the ordinary temp., gives 4(or 5)-glyoxalinemethylchloromalonamide; conc. aq. NH<sub>3</sub>, at 110°, decomp. the ester into brown, resinous products. At 110°, during 3 hrs., aq. NH<sub>2</sub> (d. 0.880) converts dl-α-chloro-β-glyoxaline-4(or 5)-propionic acid into dl-histidine (III), (decomp. 287° (cor.) not 253°) which was fully identified by the preparation of several salts. The dihydrochloride, C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>N<sub>3</sub>.2HCl, m. 235–6° (cor.) not 220°, or 225°. dl-Histidine

d-camphorsulfonate and d-tartrate, are readily sol. in H<sub>2</sub>O and crystallizes with difficulty. L-Histidine d-hydrogen tartrate, CaHaOaN. CaHaOa, large, clear, colorless, well defined prisms, or triangular plates with beveled edges, decomp.  $172-3^{\circ}$  (cor.); in  $H_1O$ ,  $[\alpha]_0$ 17.2° -16.3. The salt is more readily sol. in H<sub>2</sub>O than the d-isomer (see below). From d-tartaric acid and dl-histidine, in H<sub>2</sub>O, d-histidine d-hydrogen tartrate separates; clusters of small prisms from H<sub>2</sub>O, decomp. 234° (cor.). By means of HgCl<sub>2</sub> and Na.CO, d-histidine was regenerated from the salt; colorless, monoclinic, elongated, hexagonal plates from  $H_2O$ , decomp. 287-8° (cor.); in  $H_2O$ ,  $[\alpha]_D$  39.3°. The mother liquor from the above acid tartrate deposits l-histidine d-hydrogen tartrate. l-Histidine l-hydrogen tartrate was prepared from "natural" histidine and also from the preceding salt; clusters of prisms from  $H_2O_1$ , decomp. 234° (cor.); in  $H_2O_1$ ,  $[a]_0 - 12.1°$ . Synthetic l-histidine decomp. 287-8° (cor.); in H<sub>2</sub>O, [\alpha] = 38.1°. dl-\alpha-Hydroxy-\betaglyoxaline-4(or 5)-propionic acid, C.H.N.CH,CH(OH)CO,H, is formed from AgOH and the chloro acid (see above), in H<sub>2</sub>O; pri ms with 1 H<sub>2</sub>O, from H<sub>2</sub>O, m. 222° (cor.). The optically active isomer of this acid, corresponding with l-histidine, is described in the literature under the name "oxydeaminohistidine." At the ordinary temp., conc., aq. NH2, alc. and Et 4(or 5)-glyoxalinemethylchloromalonate hydrochloride give 4(or 5)-glyoxalinemethylchloromalonamide, C<sub>2</sub>H<sub>2</sub>N<sub>2</sub>CH<sub>2</sub>CCl(CONH<sub>2</sub>)<sub>2</sub> (see above); amorphous. Hydrochloride, C7H2O2N4Cl.2HCl, long, clear, buff spikes from dil., alc. HCl, darkens 240°, decomp. 245° (cor.). The steps by which the synthesis of histidine has been effected are as follows: citric acid --> acetonedicarboxylic acid --> diisonitrosoacetone --> diaminoacetone hydrochloride --> 2-thiol-4(or 5)-aminomethylglyoxaline --> 4(or 5)-hydroxymethylglyoxaline --> 4(or 5)-chloromethylglyoxaline hydrochloride -> ethyl 4(or 5)-glyoxalinemethylchloromalonate -> dl- $\alpha$ -chloro- $\beta$ -glyoxaline-4(or 5)-propionic acid  $\longrightarrow dl$ -histidine  $\longrightarrow l$ - (and d-) I. BISHOP TINGLE. histidine.

Equilibrium in the System: Ethyl Alcohol, Acetic Acid, Ethyl Acetate and Water, and its Apparent Displacement by Hydrogen Chloride. WILLIAM JACOB JONES AND ARTHUR LAPWORTH. Chem. Lab., Univ. Manchester. J. Chem. Soc., 99, 1427-32; Proc. Chem. Soc., 27, 143.—Measurements have been made of the total quantities of alc., AcOH, AcOEt and H<sub>2</sub>O, in equil. in presence of varying proportions of HCl, at 25° (±0.02°), the plan adopted being to allow mixtures of AcOEt with aq. HCl,

of varying conc., to remain during periods of different lengths. No appreciable amt. of EtCl was formed under the conditions employed. The equil. constant **K** = [H<sub>2</sub>O][AcOEt] is known to be about 4, over a very wide range of temp. and the [EtOH][AcOH]

value is but slightly affected by the presence of small quantities of catalysts, by alterations in the relative amts. of the constituents, or by the use of various indifferent solvents. In the author's expts., the apparent value for the equil. const.,  $\phi$ , varies from 4 to over 8, as the conc. of the HCl increases. The apparent diminution in the active mass of the H<sub>2</sub>O corresponds nearly with the formation of a compd. HCl.2H<sub>2</sub>O, or of compds. having this mean comp. The results are tabulated and are expressed graphically by means of a curve.

J. BISHOP TINGLE.

Synthesis of Oxyberberine. AME PICTET AND A. GAMS. Org. Lab., Univ. Geneva. Ber., 44, 2036-45; see C. A., 5, 3413.—N-o-Nitrobenzoyltetrakydroiso-quinoline formula (I) below, is prepared from tetrahydroisoquinoline, o-nitrobenzoyl

$$C_{\mathfrak{g}}H_{\mathfrak{g}} \subset C_{\mathfrak{g}}H_{\mathfrak{g}} \cap C_{\mathfrak{g}}H_{$$

chloride and excess of conc., aq. KOH; colorless needles from alc., m.  $75-6^{\circ}$ . Yield, 150% of the tetrahydroisoquinoline. When mixed with Me opianate (1 pt.) and conc.  $H_2SO_4$  (3-4 pts.) and allowed to remain during 2 wks., at the ordinary temp., it gives a compound (II); reddish yellow, cryst. powder from  $C_0H_0$  + petroleum ether, m.  $110-2^{\circ}$ . Yield, 100% of (I). At  $140-50^{\circ}$ , alc. KOH (15%) hydrolyzes (II) to onitrobenzoic acid and a compound (III), which are septle by means of aq.  $Na_2CO_3$ ;

slender, colorless needles from dil. alc., m. 174°. Yield, 25% of (II). It gives a violet color with conc.  $H_2SO_4$  and a trace of HNO<sub>2</sub>. Methylene-6,7-dioxytetrahydroisoquinoline (norhydrohydrastinine) (IV), is prepared from homopiperonylamine hydrochloride,  $CH_2O_2: C_6H_2CH_2CH_2NH_2.HCl$ , conc. HCl and methylal (see preceding abstr.) and is purified by means of the nitrosoamine; colorless oil with a pleasant, aromatic odor,  $b_{50}$  197-9°. Yield, about 20% of the parent amine. Hydrochloride, colorless plates from  $Et_2O +$  alc., m. 255-7°. Chloroplatinate, small, pale red prisms, m. 218-20°. o-Nitrobenzoyl derivative, is prepared in a similar manner to (I); colorless needles from dil. alc., m. 104°. Yield, 150% of the hydrochloride. Under conditions similar to those described above for (II), it condenses with Me opianate forming a compound (V); brick-red powder from  $C_6H_6 +$  petroleum ether, m. 156-8°. Yield, about 66%

of the nitrobenzoyl deriv. At 140-50°, during 2 hrs., alc. KOH (15-20%) hydrolyzes (V) to oxyberbe ine (VI), which was fully identified. Yield, 20% of (V). Oxyber-

berine, from berberine, is golden yellow, but the color is discharged by warming with Zn dust and glacial AcOH; it is therefore, due to an impurity. The synthetical product is colorless.

J. BISHOP TINGLE.

Formation of Isoquinoline Derivatives by the Action of Methylal on Phenylethylamine, Phenylalanine and Tyrosine. And Picter and Theod. Spengler. Org. Lab., Univ. Geneva. Ber., 44, 2030-6.—Tetrahydroisoquinoline is formed by digesting on the H<sub>2</sub>O-bath a mixture made by adding methylal gradually to ω-phenylethylamine, PhCH CH<sub>2</sub>NH<sub>2</sub>, in conc. HCl (about 6 pts.). Yield, about 40% of the amine. Tetrahydroisoqu noline-3-carboxylic acid, formula (I) below, is prepared in a

similar manner from d,l-phenylalanine; large, colorless, pearly lustrous scales from dil. alc., m. 311° (gas evolution). Yield, 40% of the phenylalanine. When heated above its m. p. it is decomp. into CO<sub>3</sub> and tetrahydroisoquinoline. 7-Hydroxyletrahydroisoquinoline-3-carboxylic acid (II), is obtained from d,l-tyrosine, in a similar manner to (I) and is purified by means of aq. NH<sub>3</sub>, followed by AcOH; white, insol. and microcryst., m. 336-8° (decomp.). Yield, 75% of the tyrosine. In air (II) soon decomp. Above its m. p. (II) is resolved into CO<sub>3</sub> and 7-hydroxyletrahydroisoquinoline; thick oil, b<sub>18</sub> 210-20°. It has the properties of a phenol and of a secondary amine and exhibits a violet blue fluorescence, in alc. Picrate, m. 198-201°. Hydrochloride, gives isoquinoline when distilled with Zn dust. The authors think that alkaloids of the isoquinoline series may be produced in plants by condensations similar to those described above.

J. BISHOP TINGLE.

Two New Forms of 2-Nitro-6-hydroxylaminotoluene. K. Brand. Phys. Chem. Lab., Giessen. Ber., 44, 2045-7.—The crude 2-nitro-6-hydroxylaminotoluene, prepared as described by Brand and Zöller (C. A., 1, 2713) is deposited from b. C<sub>e</sub>H<sub>e</sub> in slender, yellow needles, which change after some time to stable, pale rose-colored, opaque crystals. Both substances have the composition and properties of nitrohydroxylaminotoluene. The modification described previously consisted of opaque, stable, yellow crystals. The yellow needles are deposited always when a hot C<sub>e</sub>H<sub>e</sub> soln. is cooled quickly and they pass into both of the stable modifications spontaneously, especially in contact with a solvent, such as C<sub>e</sub>H<sub>e</sub>. The 2 stable forms are mutually transformable, by means of the unstable, yellow modification and, from hot solns. of either stable form, crystals of each stable form may be obtained by inoculation and slow cooling. The stable, yellow form, m. 117-7.5°. The rose-colored modification is transformed into it at about 105°; mixtures of the 2 stable forms, m. 114.5-5°.

Determination of Active Hydrogen in Organic Molecules. Bernardo Oddo. Univ. In t. Gen. Chem., Pavia. Ber., 44, 2048-52.—The process differs from that described by Sudborough and Hibbert (J. Chem. Soc., 85, 933) and by Zerevitinov (C. A., 5, 1285) in the following details. EtMgI is used in place of MeMgI and the

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C<sub>2</sub>H<sub>a</sub>, which is ultimately formed, is detd. by the loss in wt. of the app. instead of by measuring the CH.. Isoamyl ether and EtI are heated with excess of Mg, in a round bottomed 50 cc. flask (B), which is attached to a reflux condenser; the other end of the condenser is protected by conc. H2SO4. The substance under investigation is weighed into a second, flat bottomed flask (A) and is dissolved in anhydrous pyridine, isoamyl ether, anisole, toluene, or ligroin (b. p. 140-50°). A tube bent twice at a right angle connects (A) and (B), the shorter end projects just below the rubber stopper of (A) and the other end dips to the bottom of (B). A bulb tube containing firmly packed glass wool, followed by absorption bulbs containing conc. H<sub>2</sub>SO<sub>4</sub>, is also attached to (B). When the EtI has all reacted with the Mg (B) is quickly attached to (A) and to the absorption bulbs and the apparatus is weighed; it should not exceed 80-90 g. The absorption bulbs are connected with a U-tube filled with KOH and CaCl, and this with a flask containing alkaline soln. of pyrogallic acid. The tightness of the connections is tested by sucking air out of the pyrogallic acid flask, so that liquid rizes in the tube between (B) and (A). More air is now drawn out so that liquid passes from (B) to (A). When evolution of gas is completed, (A) is shaken and the process repeated as often as is necessary, until the evolution of C.H. ceases. The apparatus is allowed to come to equil. and is then disconnected from the U-tube and weighed. When pyridine is used in (A) it may be necessary to dip the flask in cold H<sub>2</sub>O, but with the other solvents it is often desirable to warm the liquid up to 50-60°. The quantity of materials employed should be sufficient to liberate about o.1 g. of C.H. The number of OH groups in the mol. of the substance under investigation is given by the formula  $y = (CD/P_{30.04})$ ; the % of OH =  $x(100C_{17.008}/_{30.04}P)$  = 56.6178(C/P), where 17.008 = OH, 30.04 = mol. wt. of  $C_2H_{40}$ , 56.6178 is the product of the 3 constants, C is the quantity of C<sub>2</sub>H<sub>6</sub> formed, P the wt. of substance taken and D its mols. wt. The method fails in the case of a number of aliphatic acids. The majority of the other compds. which were tested were alcs., or phenols. The error in the number of OH groups found may be  $\pm 0.21$ . J. BISHOP TINGLE.

Cyanocyclaminans [Cyanodihydrocyclamines]. III. ADOLF KAUFMANN AND ALBERTO ALBERTINI. Lab., Univ. Geneva. Ber., 44, 2052-8; see C. A., 4, 210.—9-Methyacridine picrate m. 213-4°, not 220-1° as stated by Decker, and it contains EtOH. Mercuric chloride, C<sub>14</sub> H<sub>11</sub>N.3HgCl<sub>2</sub>, yellow needles from alc., m. 258°. Methiodide, decomp. 200°, m. 235-45°, not 185° (Bernthsen). Methochloride, C<sub>15</sub>H<sub>14</sub>NCl-3N<sub>2</sub>O, from Me<sub>2</sub>SO<sub>4</sub>, followed by NaCl; yellow, lustrous plates m. about 200°. It gives a chloroplatinate, yellow and crystallin, m. 252-5°. 9,10-Dimethyl-9-cyanodihydroacridine formula (I) below, is prepared from the above methochloride and KCN,

$$C_{\bullet}H \underbrace{ \begin{pmatrix} CMe(CN) \\ NMe \end{pmatrix} } C_{\bullet}H_{4} \qquad C_{\bullet}H \underbrace{ \begin{pmatrix} C(CH_{2}Ph)(CN) \\ NMe \end{pmatrix} } C_{\bullet}H_{4}$$

in presence of Et<sub>2</sub>O, the product being purified by means of dil. HCl; colorless crystals from alc., m. 123°. Yield, 50%. *Picrate*, dark brown plates from alc., m. 138-9°. 10-Methyl-9-benzyl-9-cyanodihydroacridine (II), is prepared from 9-benzylacridine, in a similar manner to (I); white needles from alc., m. 125°. Both (I) and (II) are extremely stable and may be recrystallized from mineral acids. At high temperatures, under pressure, HCN is eliminated. Alc. KOH, at 130-40°, converts 10-methyl-9-phenyl-9-cyanodihydroacridine into 10-methyl-9-phenyldihydroacridine. At 160-

$$C_6H_4$$
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70°, during 10 hrs., alc. KOH hydrolyzes (I) to 9,10-dimethyldihydroacridine-9-carboxylic acid (III); white cubes from C<sub>0</sub>H<sub>0</sub>, white, lustrous plates from alc., darkens 130°, m. amd evolves CO<sub>2</sub> 160°. 9,10-Dimethyldihydroacridine (IV), is formed together with (III) and also from it, by heating with H<sub>2</sub>O. It is separated by its insolubility in dil. alc. KOH and is identical with the product from acridine methiodide and MeMgI (C. A., 3, 2305) (see following abstr.).

J. BISHOP TINGLE.

Cyanocyclaminans [Cyanodihydrocyclamines]. IV. Synthesis of Cinchonic Acids. ADOLF KAUFMANN AND ROBERT WIDMER. Lab., Univ. Geneva. Ber., 44, 2058-65; see preceding abstr.—N-Methyl-4-cyanodihydroquinoline formula (I) below, is oxi-

dized by I, AcONa and alc. to  $\gamma$ -cyano-N-methylquinolinium iodide (II); red needles, darkens 180°, m. 216° (decomp.). It is oxidized by alkaline  $K_aFe(CN)_a$  to N-methyl4-cyano-2-quinolone. At 150°, during 5 hrs., conc. HCl hydrolyzes (II) to  $\gamma$ -carboxy-N-methylquinolinium iodide (III). 10-Methyl-9-phenyl-9-cyanodihydroacridine does not react with I, in CHCl<sub>a</sub>, but with Br it gives a dibromo compound; white cubes from  $C_aH_a$ , m. 208-9°. It is suggested that this substance is probably 10-methyl-9-phenyl-9-cyano-3,6-dibromodihydroacridine (IV). Under similar conditions, 10-methyl-9-phenyl-9-cyano-3,6-dibromodihydroacridine (IV).

phenyldihydroacridine gives 10-methyl-9-phenyl-3,6-dibromodihydroacridinium bromide (V); orange-yellow needles from dil. alc., m. 274° (decomp.). B., aq. KCN converts it into (IV). With alc. KOH, (V) gives 9-phenyl-10-methyl-3,6-dibromodihydroacridyl ethyl ether, C<sub>12</sub>H<sub>19</sub>ONBr<sub>3</sub>; yellow needles from alc., becomes violet 170°, m. 192-5° (decomp.). 9,10-Dimethyl-9-cyano-3,6-dibromodihydroacridine, C<sub>16</sub>H<sub>19</sub>N<sub>3</sub>Br<sub>3</sub>, is prepared from Br and 9,10-dimethyl-9-cyanodihydroacridine; refractive cubes from C<sub>3</sub>H<sub>4</sub>, darkens 220°, m. 228°. The position of the Br ats. in the last 4 compds. appears to be purely hypothetical.

Cyanopinacolin and its Derivatives. OSKAR WIDMAN AND E. WAHLBERG. Lab., Univ. Upsala. Ber., 44, 2065-71.-w-Bromopinacolin, C.H.10Br, is best prepared by dissolving pinacolin (1.2 mol.), in CHCl, (20 pts.) and adding a few drops of a soln. of Br (1 mol.), in CHCl<sub>2</sub> (3-4 pts.), containing a little SnCl<sub>4</sub>. When the color has been discharged the liquid is cooled to -15° and the remainder of the Br soln. added; colorless liquid, b. 184-8° (slight decomp.); b<sub>15</sub> 77-8°; d<sub>17</sub> 1.330. The vapor attacks the eyes and skin violently. Yield, not stated; some  $\omega$ -dibromopinacolin is formed simultaneously and is separated by distillation. Tert.-valerylacetonitrile, Me,CCOCH,CN, is prepared from w-bromopinacolin, in alc. and aq. KCN, the K salt being acidified with HCl; long prisms from Et<sub>2</sub>O, m. 68-8.5°; mol. wt., in freezing C<sub>6</sub>H<sub>6</sub>, 124.1-75.3. It is volatil with steam. Yield, 76-80%. Potassium salt, Me<sub>2</sub>CC(OK): CHCN, colorless scales from AcOEt. In abs. alc. + abs. Et,O, at oo, dry HCl converts the nitrile into tert.-valerylethoxyacetimine hydrochloride, Me<sub>2</sub>CCOCH<sub>2</sub>C(OEt): NH.HCl; large, quadratic, or hexagonal prisms, m. 126-7° when quickly heated, m. 131° when slowly heated (gas evolution). Cold H<sub>2</sub>O hydrolyzes it to Et valerylacet ate. Chloroplatinate, (C<sub>2</sub>H<sub>17</sub>O<sub>2</sub>N)<sub>2</sub>.H<sub>2</sub>PtCl<sub>4</sub>.2H<sub>2</sub>O, long, yellow needles, m. 135°. Tert.-valerylacetamide, Me, CCOCH, CONH, is formed, together with EtCl, by heating the imine hydrochloride at 120-30°; large lustrous plates from C<sub>0</sub>H<sub>4</sub>, m. 95°. It gives a violet color with FeCl<sub>2</sub>. The amide is also formed from the nitrile and conc. H<sub>2</sub>SO<sub>4</sub> (9 pts.), at the ordinary temp., during 2-3 ds. *Phthalimidopinacolin*, C<sub>14</sub>H<sub>15</sub>O<sub>2</sub>N, is prepared by warming ω-bromopinacolin with K phthalimide, in alc. (90%, not abs.); lustrous, thick, quadratic prisms with hemihedral faces, m. 102°. *Pinacolylphthalamidic acid*, C<sub>14</sub>H<sub>17</sub>O<sub>4</sub>N, is formed by warming the preceding compd. with dil. aq. KOH; well developed, quadratic, or hexagonal plates, or prisms, m. 132°. Evapn. with HCl hydrolyzes it to *pinacolylamine hydrochloride*, Me<sub>3</sub>CCOCH<sub>2</sub>NH<sub>2</sub>,HCl; deliquescent.

With aq. KOCN it gives tert.-butylhydroxyimineazole, Me $_{i}$ CC  $\parallel$  ; needles, NH.COH

m. and sublimes 277-8°. The use of 90% alc. for the preparation of phthalimide derivs, appears to be of general application. The ketonic formulas given to some of the above compds, are clearly incorrect, because, as stated above, they give colors with FeCl<sub>2</sub> (see following abstr.).

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Tert .- Valerylacetic Ester. ERIK WAHLBERG. Lab., Univ. Upsala. Ber., 44, 2071-6; see preceding abstr.—Ethyl tert.-valerylacetate, CaH1aO2, is prepared by the action of H<sub>2</sub>O, at 50-60°, on tert.-valerylethoxyacetimine hydrochloride; colorless liquid w th a characteristic odor, b<sub>1</sub>, 96-7°; d<sup>18</sup>, 0.967. It is sol. in alkalies and gives an intense violet color with FeCl<sub>2</sub>. Aq. KOH (30%) hydrolyzes the ester to tert.valerylacetic acid, Me<sub>2</sub>CCOCH<sub>2</sub>CO<sub>2</sub>H; crystals from ligroin, m. 47-9°. It gives a violet color with FeCl, and, at the ordinary temp., slowly change to a liquid. At about 100° it decomp. into CO, and pinacolin. Ethyl tert.-valerylmethylacetate, C10H18O3. from the ester, MeI and EtONa; oil, b<sub>18</sub> 93-4°; d<sup>18</sup> 0.955. Aq. KOH (30%) hydrolyzes it slowly, at the ordinary temp., to tert.-valerylmethylacetic acid, CaH12O2; small, lustrous sca'es, m. 100-1° and decomp. into CO2 and tert.-butyl ethyl ketone. It gives no color with FeCl<sub>2</sub>. When warmed on the H<sub>2</sub>O bath, Et tert.-valerylacetate and PhN<sub>2</sub>H<sub>2</sub> give 1-phenyl-3-tert.-butyl-5-pyrazolone, C<sub>12</sub>H<sub>16</sub>ON<sub>2</sub>; large, colorless plates from C<sub>2</sub>H<sub>a</sub>, m. 110.5-1.5°. It is oxidized by FeCl<sub>3</sub>, in warm alc., to bis-[phenyltert.butylpyrazolone], CmH2002N4; white solid, m. above 290°. It gives a bluish green ppt. with NaNO, and dil. H.SO. 1-Phenyl-3-tert.-butyl-4-methyl-5-pyrazolone, C14-H<sub>12</sub>ON<sub>2</sub>, is prepared from Et tert.-valerylmethylacetate and PhN<sub>2</sub>H<sub>2</sub>, in the manner described above, except that the mixture is heated finally at 140°, during 6 hrs.; crystals from MeOH, m. 114.5-5.5°. Ethyl pentamethylacetoacetate, C11H20O2, is formed from Et tert.-valerylmethylacetate, MeI and EtONa, which are allowed to react, in the manner described above, during 2-3 dys.; liquid, b<sub>18</sub> 98.5-9°. When b. during 5 dys. with dil. H<sub>2</sub>SO<sub>4</sub>, it is hydrolyzed to pentamethylacetone, which was not completely purified. After heating with PhN<sub>2</sub>H<sub>2</sub>, during 8 hrs., a crystallin substance was obtained, which, in part, decomp. when recryst. from MeOH, m. 107-8°. It does not dissolve in alkali. J. BISHOP TINGLE.

Esters and Amides of Phosphoric Acids. II. Attempts to Prepare Compounds Allied to Lecithins. K. Langheld. Chem. Inst., Univs. Breslau and Würzburg. Ber., 44, 2076–87; see C. A., 4, 2806.—Ethyl metaphosphate, EtPO<sub>3</sub>, is prepared from AgPO<sub>3</sub> and EtI, in dry CHCl<sub>3</sub>; water-clear syrup which could not be distilled, it was purified by means of CHCl<sub>3</sub> + Et<sub>2</sub>O. Yield, nearly 50% of the Ag salt. It is also obtained by b., during about 60 hrs., a mixture of  $P_4O_{10}$  and excess of Et<sub>2</sub>O, dried over Na; the b. is contained until the  $P_4O_{10}$  becomes liquid. Yield, about 50% of the  $P_4O_{10}$ . Di-Et orthophosphate, purified by means of the Ba salt, is converted into the above Et metaphosphate by heating at about 80°, in a vacuum. Some alc. distils over and tri-Et orthophosphate is formed. Et metaphosphate is converted into a mixture of Et and di-Et orthophosphates by b. with abs. alc.; the Et ester is separated

by means of its Ba salts. Excess of H.O. at the ordinary temp., converts Et metaphosphate into diethyl pyrophosphate, which was isolated as the barium salt, Et.PO,Ba; amorphous. When b. with H<sub>2</sub>O the Et metaphosphate gives Et orthophosphate. After 3-4 dys. at the ordinary temp., EtPO, is converted by abs. alc. into di-Et orthophosphate. With ethylenechlorohydrin the product consists of ethyl chloroethyl orthophosphate, which was separated as the barium salt, (EtPO<sub>4</sub>C<sub>4</sub>H<sub>4</sub>Cl)<sub>2</sub>Ba. Some barium chloroethyl orthophosphate, C.H.CIPO.Ba, is also formed if the chlorohydrin is warmed with the EtPO, on the H.O-bath. Barium benzyl orthophosphate, C.H.PO,Ba.2H.O. and barium ethylbensyl orthophosphate, (C.H., PO.), Ba, are prepared together in a similar manner, from benzyl alc. and are separated by extraction with b. alc., in which the benzyl salt is sol. Ethyl choline phosphate chloroplatinate was prepared by exactly pptg. the Ba from Et chloroethyl barium phosphate, allowing the clear soln. to evap. under reduced pressure, at a low temp. and b. with alc. Me, N, the product being treated with HCl and PtCla; yellow and amorphous. Barium glyceryl phosphate, C.H.O.PBa.-0.5H<sub>2</sub>O, from dry glycerol and EtPO<sub>2</sub>. Allyl chloroethyl barium phosphate, (C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>-CIP), Ba, from AgPO,, allyl iodide, ethylenechlorohydrin and CHCl, crystals from alc. It decolorizes "permanganate" and adds Br. The following compds. were prepared by adding NH<sub>2</sub>, or the resp. amines to EtPO<sub>2</sub>, in CHCl<sub>2</sub>. They are vitreous solids, stable towards alc. HCl, but readily resolved into their components by alkali. Ammonia compound, CaH10OaN2P2. Ethylamine compound, C10H21OaN2P2. Diethylamine compound, C10H21O2N2P. Triethylamine compound, C18H2OO2NP. Carbamide compound, C.H.O.N.P., syrup. The following are also vitreous: alanine compound, C7H17O2NP2. Valine compound, C2H21O2NP2. Leucine compound, C10H22O2NP2, it gives with Et,NH the salt, C22H26O2N4P2. Aspartic acid compound, C2H12O7NP. Glutamic acid compound, C<sub>2</sub>H<sub>16</sub>O<sub>7</sub>NP. Serine compound, C<sub>2</sub>H<sub>20</sub>O<sub>18</sub>NP<sub>2</sub>, its state of aggregation is not mentioned. The above derivs. of primary amines are regarded as being imido-orthophosphoric esters, (EtOPOOH), NR (R = H or an alkyl). The preceding compds. formed from secondary amines, or from primary amines containing a strongly acidic group, are formulated as amidoorthophosphoric esters, EtOPO-(OH)NRR' (R' may be H). With tertiary bases the products are supposed to be

cyclic esters, OP(OEt)

O
NR<sub>s</sub>

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Formation of Cycloids in the Peri Position in the Naphthalene Series. IV. tempts to Produce a Carbon Ring of Six Members. Franz Sachs and Percy Brigl. Chem. Inst., Univ. Berlin. Ber., 44, 2091-106.—Accompanying the 2-hydroxy-1naphthaldehyde, HOC<sub>10</sub>H<sub>4</sub>CHO, prepared from β-naphthol, HCN, HCl, ZnCl<sub>2</sub> and Et<sub>2</sub>O is a substance, probably 2-hydroxyhydronaphthamide, (HOC<sub>10</sub>H<sub>0</sub>CH)<sub>2</sub>N<sub>2</sub>, which is separated by its insolubility in alc.; slender, microscopic, bright red needles from PhNO, m. 312°. At the b. p. it dissolves in 150 pts. of PhNO<sub>2</sub>. Yield, about 3%. The compd. is hydrolyzed by b., conc. HCl to NH, and 2-hydroxy-1-naphthaldehyde. The yield of 2-ethoxy-1-naphthaldehyde, from the hydroxy deriv., is only 50% by the use of EtBr, but with EtSO4 it is 91%. In alc., with PhNH2, it forms 2-ethoxy-1-naphthaldehydeanil, EtOC, oH, CH: NPh; acute-angled plates from alc., m. 73°. Phenylhydrazone, EtOC<sub>10</sub>H<sub>e</sub>CH: NNHPh, prepared in warm, glacial AcOH; light yellow plates from glacial AcOH, m. 91°. It is unstable in air. Semicarbazone, EtOC, 10HaCH: NNHCONH<sub>4</sub>, long, white needles from MeOH, m. 214-5°. It is the most characteristic deriv. of the aldehyde. 2-Ethoxy-1-naphthylhydroxyacetonitrile, EtOC10H0CH(OH)CN, from the aldehyde and anhydrous HCN, at the ordinary temp.; large plates. It is resolved into its components by soln. in Et<sub>2</sub>O. 2-Ethoxy-1-naphthaldehyde does not condense with AcOEt, in presence of Na, nor with malonic ester, in presence of piperidine, but with malonic acid and PhNH<sub>2</sub> it gives naphthocoumarinearboxylic acid, CH: CCO<sub>2</sub>H

CH: CCO<sub>2</sub>H C<sub>10</sub>H<sub>6</sub> | Yield, 71%. At 145°, aq. NH<sub>2</sub> (25%) and (NH<sub>4</sub>)<sub>2</sub>SO<sub>2</sub> con-

vert 2-hydroxy-1-naphthaldehyde into β-naphthylamine. At 193-205°, PCl<sub>a</sub> and the hydroxy aldehyde give 2-chloro-1-dichloromethylnaphthalene, ClC10HaCHCl2; rhombic plates from alc., m. 90°. Yield, 70-4%. The PCl<sub>s</sub> is allowed to react in closed tubes, which must not contain more than 6 g. of the aldehyde for each 100 cc. of their capacity. At 100°, MeONa + MeOH convert the preceding compd. into 2-chloro-1-naphthaldehydedimethylacetal, ClC10H6CH(OMe)2; white, acute-angled prisms from abs. alc., m. 86°. Yield, 82%. 2-Chloro-1-naphthaldehyde, ClC<sub>10</sub>N<sub>6</sub>CHO, is best prepared from the above trichloro deriv. and b., dil. AcOH; long, soft, white needles from alc., m. 76°. It is volatil with steam. Yield, 93%. Azine, ClC10HaCH: NN: CH-C<sub>10</sub>H<sub>a</sub>Cl, from the aldehyde and N<sub>a</sub>H<sub>a</sub>.H<sub>a</sub>O; opaque, golden yellow needles from BzOEt, m. 195°. Yield, 90%. Semicarbazone, slender, microscopic needles from abs. alc., m. 215°. Yield, 85%. The only serviceable method which could be discovered for the conversion of the chloroaldehyde into 2-chloro-1-naphthylacrylic acid, ClC<sub>10</sub>H<sub>a</sub>CH: CHCO<sub>2</sub>H, consisted in heating the aldehyde (1 g.) with anhydrous AcOK (1 g.) and recently distilled Ac<sub>4</sub>O (5 cc.), at 170°, during 3.75 hrs.; long needles from AcOH (75%), m. 176°. Yield, 57%. Ammonium salt, crystallin. Calcium salt, amorphous, white powder. Silver salt, unstable to light. No chloride could be isolated, but the product from the acid and PCla gives an amide, C12HaClCONHa; white, opaque needles from abs. alc., m. 195°. Yield, 75%. Attempts to reduce the acid were either unsuccessful, or they resulted in the simultaneous elimination of Cl. Dehydrating agents either decomp. the acid completely, or they fail to react with it. Bis-2-chloro-1methylnaphthalacetone, (ClC<sub>10</sub>H<sub>a</sub>CH: CH)<sub>2</sub>CO, is prepared from the chloroaldehyde, acetone and conc. H<sub>2</sub>SO<sub>4</sub>, at o°; canary-yellow needles from BzOEt, m. 215°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is indigo blue, it is discharged by adding H<sub>2</sub>O. 2-Chloro-1methylnaphthalmethyl ethyl ketone, ClC<sub>10</sub>H<sub>a</sub>CH: CHCOEt, from the aldehyde, methyl ethyl ketone, KOH and dil. alc., first at the ordinary temp., then at oo; white, soft needles from alc., m. 74°. Yield, 77%. In conc. H<sub>2</sub>SO<sub>4</sub> the color is reddish brown and it is discharged almost completely by adding HNO<sub>3</sub>. 2-Chloro-1-naphthaldehyde gives a pale yellow color with conc. H2SO4, changing to blood-red with a drop of HNO2. Semicarbazone, C18H18ON,Cl, silky lustrous, white needles from MeOH, m. 185°. Yield, 80%. ω-Hydroxy-ω-[2-chloro-1-naphthyl]-diethyl ketone, ClC<sub>10</sub>H<sub>6</sub>CH(OH)CH<sub>2</sub>COEt, is prepared in a similar manner to the preceding ketone, except that only enough aq. KOH is added to produce a feeble alkaline reaction with litmus; double pyramides with acute angles and pointed ends from ligroin, m. 124°. Yield, 76%. w-[2-Chloro-1-naphthyl] diethyl ketone, ClC10H6CH2CH2COEt, is prepared by reducing the above unsaturated naphthal ketone with Al-Hg; oil. It could not be distilled, it is not volatil with steam and it could not be condensed to a cyclic compd. With semicarbazine it gives a compound, C10H15ON2Cl, which has no basic properties. J. B. T.

Dihydroquinaldine Bases. Gustav Heller. E. Beckmann's Lab. Appl. Chem., Leipzig. Ber., 44, 2106–15; see C. A., 2, 3352.—Dihydroquinaldine is best prepared by b. quinaldine (5-10 g.) with conc. HCl (10 pts.), H<sub>2</sub>O (10 pts.) and Zn dust, in excess; when evolution of H ceases an equal vol. of H<sub>2</sub>O is added and some AcONa. It is purified by extracting with alc. and pptg. with H<sub>2</sub>O. Yield, 95%. Hydrochloride, from the amine in alc. and warm HCl; lustrous needles, darkens 210°, m. about 250° (decomp.). Sulfate, lustrous plates resembling naphthalene, darkens about 210°, decomp. about 240°. Dihydroquinaldine is oxidized to quinaldine slowly by air, more rapidly by yellow HgO, or CrO<sub>2</sub>; it does not combine with MeI and does not

form a nitroso compd. With Br, in CHCla, it gives dibromodihydroquinaldine, CasHa-NBr<sub>2</sub>; colorless, lustrous needles from toluene, m. 214°; after heating with HgO, in cumene, m. and darkens 242°. Yield, 75% It is not changed by MeI, at 100°, but at 160° complete decomp. takes place. β-o-Nitrophenyl-a,β-dibromoethyl methyl ketone. O.NC.H.CHBrCHBrAc, is prepared from o-nitrocinnamyl methyl ketone and Br, in glacial AcOH; colorless needles from ligroin, m. 102°. In glacial AcOH, Zn dust reduces it to quinaldine and tetrahydroquinaldine. o-Toludihydroquinaldine. (C,,-H<sub>12</sub>N)<sub>20</sub> is prepared from o-toluquinaldine, in a manner similar to that described above for quinaldine; colorless needles from alc., m. 216-7°; mol. wt., in freezing CaHa, 267-91. Yield, 85%. No salts could be obtained; with HgO, or CrO, it regenerates o-toluquinaldine. At the ordinary temp., in CHCl, Br converts o-toludihydroquinaldine into a mixture which could not be separated, but in hot alc., or glacial AcOH, o-toludikydrotetrabromoquinaldine, C11H2NBr4, is formed; S-yellow crystals from C2H2 + ligroin, m. 171-2°. In more dil. solns. a tribromo derivative, C11H10NBr2, is produced; crystals from alc., m. 121-2°. p-Toluquinaldine is reduced, in the manner described above, to p-toludihydroquinaldine, which was purified by means of a salt; crystallin, m. 143°; mol. wt., in freezing C<sub>4</sub>H<sub>4</sub>, 321-42. It could not be reduced to the tetrahydro compd. with HgO, or CrO, it regenerates p-toluquinaldine. Yield, 36 and 65%, resp. Hydrochloride, white needles, darkens about 205°, m. p. indefinit. Hydrobromide, lustrous needles, darkens about 215°, m. about 260°. Sulfate, plates, darkens about 206°, m. about 220° (decomp.). Tetrabromo derivative, prepared like the isomer above; pale yellow needles from CHCl<sub>2</sub> + ligroin, m. 172°. m-Toludihydroquinaldine is amorphous; it is prepared like the isomers. Quinoline also gives an amorphous dihydro deriv. o-Methyldihydroquinoline, C10H11N, is obtained from o-methylquinoline, in the manner described above and is purified by means of a salt; cryst., m. 144°. Hydrockloride, from dry HCl, in abs. alc.; cryst., m. about 278°. With HgO, but not with CrO, the amine regenerates o-methylquinoline. Yield, 40%. p-Methyldihydroquinoline resembles the preceding isomer and is prepared in a similar manner; cryst., m. about 60-105°; mol. wt., in freezing C<sub>2</sub>H<sub>4</sub>, 290-302. It could not be purified completely, because it does not form salts. It regenerates p-methylquinoline with HgO, but not with CrO<sub>2</sub>. Yield, 50%. Doebner's statements regarding the properties of dihydroquinaldines are incorrect (Ber., 31, 690). J. BISHOP TINGLE.

Action of Nitric Acid on Trimethylgaliic Acid [3,4,5-Trimethoxybenzoic Acid] and its Methyl Ester; the Constitution of Antiarol. H. THOMS AND W. SIEBELING. Pharm. Inst., Univ. Berlin. Ber., 44, 2115-25 .- Methyl 3,4,5-trimethoxy-2,6-dinitrobenzoate, (O2N)2Ca(OMe)2CO2Me, is prepared from Me 3,4,5-trimethoxybenzoate and fuming HNO, in glacial AcOH, at first at oo, but finally the mixture is heated until its bloodred color changes to golden yellow; colorless needles from MeOH, m. 111°. Yield, 40%. The ester could not be nitrated further; a mixture of H<sub>2</sub>SO<sub>4</sub> + HNO<sub>2</sub> causes its complete decomp. At the ordinary temp., during 2 hrs., alc. KOH hydrolyzes the ester to 3,4,5-trimethoxy-2,6-dinitrobenzoic acid; short, colorless needles from CoHe, m. 158-60°. It becomes yellow on exposure to light. Yield, 90% of the ester. When treated with MeOH and dry HCl, it regenerates the ester with difficulty. Barium salt, pale yellow prisms; it is very readily sol. in H<sub>2</sub>O. Silver salt, long, slender, yellow, microscopic needles. 3,4,5-Trimethoxy-2 nitrobenzoic acid, O,NC,H(OMe),CO,H, is prepared from the ester and alc. KOH, on a H<sub>2</sub>O bath; colorless, acute-angled prisms from C<sub>a</sub>H<sub>e</sub>, m. 163-4°. Ammonium salt. Silver salt, pale yellow, well developed, opaque crystals from H<sub>2</sub>O. Barium salt, readily sol. in H<sub>2</sub>O. When warmed with conc. HNO, the acid gives 1,2-dinitro-3,4,5-trimethoxybenzene. At 190-220°, the acid evolves CO, and gives a compd., possibly 2-nitro-3,4,5-trimethoxybenzene. It was obtained only in very small quantity. Under similar conditions, the above dinitro acid evolves CO<sub>2</sub> quant. and forns what is probably 2,6-dinitro-3,4,5-trimethoxybenzene, (O<sub>4</sub>N)<sub>2</sub>CH(OMe)<sub>2</sub>; large, yellow, transparent crystals from MeOH + H<sub>2</sub>O, m. 85°. Conc. HNO<sub>2</sub> does not react with the dinitro acid, but when this latter is added to a cooled mixture of equal vols. of fuming HNO<sub>3</sub> and conc. N<sub>2</sub>SO<sub>4</sub>, 1,2,6-trinitro-3,4,5-trimethoxybenzene, (O<sub>4</sub>N)<sub>2</sub>C<sub>6</sub>(OMe)<sub>2</sub>, is produced; slender, yellow needles from MeOH + H<sub>2</sub>O, m. 128°. Yield, 25% of the dinitro acid. 1,2-Dinitro-3,4,5-trimethoxybenzene was prepared from 3,4,5-trimethoxybenzoic acid, from its nitro derivative and also from pyrogallyl trimethyl ether, m. 119° not 126° as stated by Will. 1-Nitro-3,4,5-trimethoxybenzene, when reduced and diazotized, gives 3,4,5-trimethoxyphenol, it is identical with "antiarol," which is contained in the milky sap of Antiaris toxicaria Leschen and also with the phenol obtained by the partial methylation of dimethoxyhydroquinol (Will).

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Products of the Action of Nitric Acid on Dihydroanethole. H. THOMS AND W. DRAUZBURG. Pharm. Inst., Univ. Berlin. Ber., 44, 2125-33.—"Dihydroanethol" is 4-methoxypropylbenzene. When warmed with an equal quantity of HNO<sub>2</sub> (45%) it gives a mixture of p-methoxybenzoic acid, p-methoxybenzaldehyde and the 2 nitro compds. described below. The acid is deposited, in part, as crystals and the remainder is extracted from the oily reaction product by means of hot H<sub>2</sub>O. The dinitro compd. is extracted from the oil by means of Na<sub>2</sub>CO<sub>2</sub> and the aldehyde by the use of NaHSO<sub>2</sub>. 2-Nitro-4-methoxypropylbenzene, O.NC.H. (OMe)Pra is purified by distillation; oil, b. 164-9°. It is oxidized to 3-nitro-4-methoxybenzoic by the action of KMnO, and H.SO. (20%). 3,5-Dinitro-4-hydroxypropylbenzene, (O,N),CaH,PraOH, prismatic rods from Et.O. m. 46°. It is volatil with steam. Sodium salt, needles with a dark reddish green luster, softens and explodes 264°. Polassium and silver salts, explode when heated. Acetyl derivative, C11H12O2N2, from Ac2O+AcONa; greenish yellow needles from ligroin, m. 89°. It does not dissolve in aq. Na<sub>2</sub>CO<sub>2</sub>, or NaOH. Benzoyl derivative, C<sub>16</sub>H<sub>14</sub>O<sub>6</sub>N<sub>2</sub>, from Bz<sub>2</sub>O, but not from BzCl; m. 86.5-7°. Diphenylurethan, (O<sub>2</sub>N)<sub>2</sub>C<sub>2</sub>H<sub>2</sub>Pr<sup>a</sup>OCONPh<sub>2</sub>, from diphenylcarbamyl chloride and pyridine; cubical crystals from ligroin, m. 136.5°. 3,5-Dinitro-4-carbomethoxypropylbenzene, (O2N)2C3H2PraOCO2Me, from the Na phenolate and Me chlorocarbonate, at --6°; almost white needles from ligroin, m. 85.5-6.5°. The phenol could not be methylated by means of MeI, or of Me<sub>2</sub>SO<sub>4</sub>. By the further action of HNO<sub>2</sub> (45%), 3-nitro-4-methoxypropylbenzene is converted into 3,5-dinitro-4-hydroxypropylbenzene. This latter compd. is reduced by heating with HCl (25%) and Sn. The resulting amine is unstable, but the following derivatives of it were prepared. 3,5-Diamino-4-hydroxypropylbenzene dihydrochloride, (H,N),C,H,Pr\*OH.2HCl; slender, white crystals, stable when dry. Diacetyl derivative, (AcNH), CaH, PraOH, is formed, at the ordinary temp., by adding Ac<sub>2</sub>O slowly to a mixture of the dihydrochloride, AcONa, H<sub>2</sub>O and AcOH; needles from alc., or H<sub>2</sub>O, m. 161.5-2°. Dibenzoyl derivative, prepared by the Schotten-Baumann method; aggregates of slender needles from alc., m. 198°. With NaNO, the amine forms a brown dye, but it could not be diazotized. These results indicate that the amine probably has the NH, groups J. BISHOP TINGLE. in the m- (3,5-) position.

Elimination of Methoxyl Groups from Phenol Ethers by means of Nascent Hydrogen. H. Thoms and W. Siebeling. Pharm. Inst., Univ. Berlin. Bet., 44, 2134-6.—1,2,3-Trimethoxybenzene is converted into resorcinyl dimethyl ether by treatment, on a H<sub>2</sub>O bath, with Na and alc. Isoeugenyl methyl ether, under similar conditions, gives a phenol in small quantity, but 4-methoxypropylbenzene, anethole, asaron, its dihydro derivative and m-propylphenyl ether are not changed. J. B. T.

Strychnine Alkaloids. XI. The Brucine-Nitric Acid Reaction and Preparation of Bisapomethylbrucine, a New Alkaloid. HERMANN LEUCHS AND RUDOLPH ANDERSON. Chem. Inst., Univ. Berlin. Ber., 44, 2136-45; see C. A., 4, 3226.—Bisapo-

methylbrucine, C<sub>11</sub>H<sub>20</sub>O<sub>4</sub>N<sub>2</sub>, is prepared by treating dried brucine (20 g.) with 5 N HNO<sub>2</sub> (100 cc.), at oo, until a clear soln. is formed, this is then mixed with H<sub>2</sub>O (100 cc.) that has been saturated with SO, at o°. The crude material is purified by means of the hydrochloride; long, hexagonal prisms with 1 EtOH from alc., darkens about 260°, decomp. 285°, not m. 305°. Its aq. soln. gives a deep indigo-blue color with Fe" salts, it is discharged by acids. Bisapomethylbrucine reduces AgNO, immediately, its aq. soln. is alkaline towards litmus and curcuma and it gives a red color with dil. HNO2 only after prolonged standing, or wnen warmed. Hydrochloride, C21H2O4N2.HCl, small, lustrous, colorless, or pale green, massive polyhedra at 20-5°, slender needles at lower temps. from dil. HCl, not changed up to 300°. A sulfate of variable comp. was also prepared. The red color which is produced when brucine and HNO, are mixed, is due to the formation of a quinone: it was isolated by allowing the alkaloid and acid to react during 0.5 hr., in the manner described above and then adding KHCO; red, or reddish brown, insol. powder, C2H2nO1N2.HNO2. Yield, 88% of the brucine. When the acid soln. is diluted with H<sub>2</sub>O, at o°, then the KHCO, added and the liquid extracted immediately with CHCl, a compound, C21H20O4N2.H2O, is obtained; dark red crystals by adding b. ligroin to the b. CHCl, soln. Yield, 68% of the brucine. At 78°, or by recryst. from b. ligroin, needles with 0.5 H<sub>4</sub>O are deposited. With HCl and HCl it gives a salt, CaH200,N2.H2CrO4.HCl, bright red, crystallin powder. It forms the quinone,  $C_n H_{20}O_4N_2$ , when mixed with dry NaCO<sub>2</sub> and treated with H<sub>2</sub>O + CHCla. The quinone resembles the hydrate closely, it is reduced by SO<sub>2</sub> to a yellow quinone hydrate, which fails to form crystallin salts. This hydrate gives cacotheline by the action of HCl + CrO<sub>2</sub>; it was identified as the nitrate, C<sub>n</sub>H<sub>2</sub>O<sub>2</sub>N<sub>2</sub>.HNO<sub>2</sub>.H<sub>2</sub>O. [The authors' application of the term "new alkaloid" to bisapomethylbrucine is, without justification; consequently the title of their paper is misleading. ABSTR.]

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New Synthesis of Trihydroxythioxanthones. FRITZ ULLMANN AND MASUO SONE. Techn. Chem. Inst. Roy. Techn. Hochsch.. Berlin. Ber., 44, 2146-8.—2,3,4-Tri-hydroxy-7-methylthioxanthone formula (I) below, is prepared by mixing 3,4,5-trihy-

droxybenzoic (gallic) acid (17.2 g.) with p-thiocresol (4 g.) and adding conc. H<sub>3</sub>SO<sub>4</sub> (80 cc.), while stirring. The mixture is eventually heated up to to°, during 1 hr.; feathery aggregates of yellow needles from alc., darkens above 240°. Its color with FeCl<sub>2</sub> is brown, with dil. alkali reddish brown and with conc. H<sub>3</sub>SO<sub>4</sub> orange, with a green fluorescence. Trimethyl ether, from Me<sub>2</sub>SO<sub>4</sub>; pale yellow needles from ligroin, m. 135°. Its soln. in C<sub>2</sub>H<sub>4</sub> is pale yellow with a bluisn green fluorescence, in conc. H<sub>3</sub>SO<sub>4</sub> orange-colored with a green fluorescence. 2,3,4-Trihydroxythioxanthone (II), is prepared from thiophenol in the manner described above and is identical with the compd. of B. G. Davis and S. Smiles (C. A., 4, 3073), from thiosalicylic acid and pyrogallol. Trimethyl ether, pale yellow needles, m. 153-4°. In alc., or C<sub>2</sub>H<sub>6</sub> the color is pale yellow, with a bluish green fluorescence. Both (I) and (II), dye wool, mordanted with Cr, olive-yellow and cotton, mordanted with Al, or Sn, yellow.

Electrolytic Reduction of Anisic Aldehyde. Julius Tafel and Wilhelm Schepss. Chem. Inst., Univ. Würzburg. Ber., 44, 2148-54.—The expts. recorded below were made in the small apparatus described previously (Ber., 33, 2216 (1900)) and also in a larger form (C. A., 3, 2567), somewhat modified. The anode liquid was

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in all cases  $H_2SO_4$  (30%). The cathode liquid, in the smaller apparatus, consisted of p-methoxybenzaldehyde (2 g.), 30%  $H_2SO_4$  (6 g.) and 96% alc. to make 20 cc. Expts. on a larger scale were made with the aldehyde (20 g.),  $H_2O$  (60 cc.), varying quantities of conc.  $H_2SO_4$  and alc. to make 180 cc. The anode and cathode compartments were heated, or cooled, as desired. The cathode was of Cd. The products of the electrolysis were extracted by means of  $Et_2O$  and fractionated, they consisted of p-methoxytoluene up to 60%, a little p-methoxybenzyl alc. and p,p'-dimethoxystilbene, together with small quantities of other high b. substances. An increase in the current density, provided that the temp. is not allowed to rize, favors the production of p-methoxytoluene. A study was made of the effect of variation in conditions on the yield of the different products. The substances which are formed are the same as those obtained by Law (C. A., 1, 173; J. Chem. Soc., 91, 748 (1907)) who used a Cu cathode, but the relative proportions are quite different. Law' yield of p-methoxytoluene, for example, was only 5% of the aldehyde.

Constitution of Alloxantin. M. M. RICHTER. Private Lab., Karlsruhe. Ber., 44, 2155-8.—Alloxan and p-phenylenediamine, in H<sub>2</sub>O, at 30°, give an additive compound formula (I) below, bluish black, decomp. without m. With N<sub>2</sub>H<sub>4</sub>.H<sub>2</sub>O, in abs. MeOH,

alloxan gives a similar compound,  $C_4H_6O_4N_4$ ; white and amorphous, decomp. without m. It is hydrolyzed gradually by  $H_2O$ , to N and  $NH_4$  dialurate. These results lead the author to conclude that the compds. in question are similar in structure to quinhydrones, for which he has suggested formulas  $(C.\ A.,\ 5,\ 1285)$ . He regards alloxantin as an oxonium compd. (II). His quinhydrone formulas have been shown to be in-

$$OC \left\langle \begin{array}{c} NH \cdot CO \\ NH \cdot CO \\ \end{array} \right\rangle C : OHOCH \left\langle \begin{array}{c} CO \cdot NH \\ CO \cdot NH \\ \end{array} \right\rangle CO$$

correct by Knorr (C. A., 5, 3070). Alloxantin is usually formulated as  $C_2H_4O_7N_4$  with 1 and 3  $H_2O$ .

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Preparation and Scission of Dihydroindole. J. v. Braun and W. Sobecki. Chem. Inst., Univ. Breslau. Ber., 44, 2158-61.—The best conditions for the electrolytic reduction of indole to dihydroindole are as follows: the cathode liquid consists of indole (15 g.), alc. (200 cc.), H<sub>2</sub>O (70 cc.) and conc. H<sub>2</sub>SO<sub>4</sub> (25 cc.); it is placed in a porous cell, connected with a reflux condenser. The anode liquid consists of H<sub>2</sub>SO<sub>4</sub> (20%). The electrodes are of Pb and a current of about 35 amperes, at 36 volts, is passed during 5 hrs., the cathode liquid being allowed to reach its b. p. The alc. and unchanged indole (about 2 g.) are separated by steam distillation, the residue is made alkaline and the dihydroindole volatilized by steam. Yield, 5.5-6 g. Phenylsulfonyl derivative, C<sub>8</sub>H<sub>8</sub>NSO<sub>2</sub>Ph, needles from alc., m. 133°. Benzoyl derivative, C<sub>8</sub>H<sub>8</sub>NBz, poorly developed crystals from alc., or from alc. + H<sub>2</sub>O, m. 118°. It is formed by the direct reduction of benzoylindole, but is best prepared from dihydroindole. At 135-40°, during 1 hr., PCl<sub>s</sub> causes a scission of the indole ring. The product, after digestion with warm H<sub>2</sub>O, consists of o-β-chloroethylbenzanilide, ClCH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>NHBz; colorless, opaque crystals from alc., m. 120°. Yield, 30%. J. BISHOP TINGLE.

Quinoids. XXV. Aniline Black. V. RICHARD WILLSTÄTTER AND CARL CRAMER. Chem. Lab. Fed. Techn. Hochsch., Zurich. Ber., 44, 2162-71; see C. A., 5, 498 and following abstrs.—This paper is essentially a reply to A. G. Green and A. E. Woodhead (C. A., 5, 1085) and a repetition of some of their work and of some of Will-

stätter's. In place of Willstätter's "monoquinoidal," "diquinoidal," etc., the English chemists have suggested the terms "leucoemeraldine" for the leucobase, C<sub>48</sub>H<sub>42</sub>N<sub>8</sub>, "protoemeraldine," "emeraldine" "nigraniline" and "pernigraniline" for the mono-, di-, tri- and tetraquinoidal stages, resp. The oxidation stages have been detd. incorrectly by Green and Woodhead. "Emeraldine" and "nigraniline" are tri- and tetraquinoidal, resp. and not di- and triquinoidal. Green and Woodhead state that the chief characteristic of tri- and tetraquinoidal "black" is their solubility in AcOH (80%) and in HCO<sub>2</sub>H (60%). This is incorrect. The small portions of the substances in question which do dissolve, undergo a change; the greater part of the material is merely in suspension. The present state of knowledge regarding the oxidation stages of aniline black is summarized.

Quinoids. XXVI. o-Quinones. RICHARD WILLSTÄTTER AND FRITZ MÜLLER. Chem. Lab. Federal Techn. Hochsch., Zurich. Ber., 44, 2171-81; see preceding and following abstrs. and C. A., 2, 3079.—When pyrocatechol is rapidly oxidized by means of Ag<sub>2</sub>O, in Et<sub>2</sub>O which has been dried as completely as possible, colorless, very unstable crystals are deposited; they are regarded as being pyrocatechyl peroxide,

C<sub>s</sub>H<sub>s</sub>(). They change quickly into the stable, red crystals of pyrocatechoquinone,

O: C<sub>n</sub>H<sub>4</sub>: O. 4-Methyl-1,2-benzoquinone, O: C<sub>n</sub>H<sub>2</sub>Me: O, is prepared in a similar manner to the preceding compd., from 4-methylpyrocatechol, in presence of Na<sub>2</sub>SO<sub>4</sub>; dark red, triclinic prisms, m. 83-4°. It is volatil under reduced pressure, at the ordinary temp, and its solns, are green to red. Like the parent phenol, it does not form a quinhydrone, but it gives the ordinary quinone reactions. Mol. wt., in freezing C<sub>6</sub>H<sub>6</sub>, 121-7; in b. Et<sub>2</sub>O, 115-141; after 1.5 hrs., 195. On 1 occasion during the preparation of the quinone, unstable, colorless crystals were obtained, they consisted, presumably, of the peroxide. When the solvent is rapidly volatilized thin, yellow prisms and needles are deposited, crystallographically they are identical with the above red, triclinic prisms. This yellow form often m. 65-7°; it is supposed to be a mixture of the white peroxide and red quinone and it is converted into the latter by exposure to light. Dimol.-4-methyl-1,2-benzoquinone, (C,H6O2)2, is prepared by the insolation of the preceding red quinone, or by the prolonged b. of its Et.O, or acetone solns.; yellow, tabular prisms of the rhombic system from CHCl<sub>3</sub> + Et<sub>2</sub>O, m. 124-5°; mol. wt., in b. acetone, 226-45. The compd. does not give the quinone reactions and from it the monomol. form could not be regenerated. 3-Methyl-1,2benzoquinone, is prepared from 3-methylpyrocatechol and could be obtained only in the colored form; long, dark red prisms, or needles and triangular plates from Et,O + petroleum ether. It is very unstable and gives the quinone reactions. Dimol. form, by warming an Et<sub>2</sub>O soln. of the quinone; yellow, monoclinic prisms and plates from CHCl + petroleum ether, m. 194-5°. It does not exhibit the quinone reactions. Pyrogallyl 1-methyl ether, b,0 129°. It gives, with Ag,O, 3-methoxy-1,2-benzoquinone, O: C<sub>6</sub>H<sub>3</sub>(OMe): O; dark red, or brownish red, rhombic plates or acute-angled prisms from Et<sub>2</sub>O, m. 115-20°. It exhibits the quinone reactions and is stable. 2-Hydroxy-1,4-quinone, O: C<sub>6</sub>H<sub>2</sub>(OH): O, is prepared from 1,2,4-trihydroxybenzene, in the manner described above; yellow, rectangular plates from C<sub>n</sub>H<sub>n</sub>, darkens 120°, chars without m. 128°. It is stable and shows the quinone reactions. With 1,2,4-trihydroxybenzene it gives a quinhydrone; black prisms with a greenish luster from Et<sub>2</sub>O. The formation of this compd. and the color of the quinone show that the latter is a p-quinone, as formulated above. J. BISHOP TINGLE.

Quinoids. XXVII. Chloro Derivatives of Pyrocatechol and of o-Quinone. RICHARD WILLSTÄTTER AND HANS EDUARD MÜLLER. Chem. Lab. Federal Techn.

Hochsch., Zurich. Ber., 44, 2182-91; see preceding abstrs.—Pyrocatechol (500 g.) and SO<sub>2</sub>Cl<sub>2</sub> (620 g.), in com. Et<sub>2</sub>O (1500 g.) when well stirred and cooled form a mixture of the Cl derivatives described below. Yield, of crude material 1492 g. 4-Chloropyrocatechol, ClC<sub>a</sub>H<sub>3</sub>(OH)<sub>2</sub>, is usually obtained as the hydrate with 0.5 H<sub>2</sub>O. This is not changed by repeated recrystallization from various anhydrous solvents, but the dehydrated comp. may be obtained either by the use of dry Et<sub>2</sub>O and the exclusion of moisture during the chlorination, or by the repeated distillation of the hydrate in a vacuum; the H<sub>2</sub>O passes over first. The anhydrous chlorophenol b<sub>8.8</sub> 136-6.5°; b<sub>10.8</sub> 139-9.5°; leaflets, or from CS, in prisms, m. 90-1°, after resolidification m. 59-61°. With FeCl, the color is green, changing to dark red with Na, CO,, to blue with AcONa and to reddish violet with AcONa in excess. Diacetyl derivative, from Ac<sub>2</sub>O + H<sub>2</sub>SO<sub>4</sub>; highly refractive oil with a slight odor, b<sub>7-8</sub> 145-7°. Dibenzoyl derivative, from BzCl + pyridine; slender needles from Et<sub>2</sub>O, m. 96-7°. Contrary to the statement of Peratoner (Gazz, chim. ital. 28, I, 197 (1898)) 3-chloropyrocatechol is formed together with the 4chloro compd. and is separated by means of its lower b. p. and greater solubility; small scales, b<sub>11</sub> 110-1°; m. 46-8°. It absorbs H<sub>2</sub>O from air and when the hydrated material is distilled under reduced pressure the crystallized distillate contains 0.5 H.O. With FeCl, the color is bluish green, changing to light red with Na<sub>2</sub>CO<sub>2</sub>, the addition of AcONa produces the same changes as with the 4-isomer. Dibenzoyl derivative, needles, m. 108-9°. 4,5-Dichloropyrocatechol, Cl<sub>2</sub>C<sub>2</sub>H<sub>2</sub>(OH), is prepared from pyrocatechol, or from its 4-chloro compd., by means of 2, or 1 mot. of SO<sub>2</sub>Cl<sub>2</sub>, resp.; opaque prisms from CHCl<sub>2</sub> and CS<sub>2</sub>, m. 116-7° (not 105-6°). It is stable in air, decomp. when warmed even in soln. and behaves towards FeCl, like the 4-chlorophenol. With H<sub>2</sub>O it forms a hydrate; lustrous, rhombohedral crystals with 0.5 H<sub>2</sub>O, decomp. when heated. From glacial AcOH, cubical crystals with I AcOH are deposited. Dibenzoyl derivative, lustrous, rhombic plates, or prisms, m. 140-0.5°. In glacial AcOH, Cl (1 mol.) converts 4,5dichloropyrocatechol into the trichloro compd. described by H. Cousin (Ann. chim. phys., [7] 13, 480 (1898)), m. 106-9°. This method of preparation shows that the compd. must be 3,4,5-trickloropyrocatechol. In addition to the monohydrate, which is already known, tetrachloropyrocatechol forms a trihydrate, C<sub>6</sub>Cl<sub>4</sub>(OH)<sub>2</sub>·3H<sub>2</sub>O, slender needles, m. 94°. It is best prepared by adding H<sub>2</sub>O, in excess, to the anhydrous phenyl, in alc. It is not changed by recrystallization from H.O. alc., or acetone, but is dehydrated by the action of C<sub>0</sub>H<sub>a</sub>, or of ligroin, or over H<sub>2</sub>SO<sub>4</sub> in a vacuum. Tetrachloropyrocatechol may be titrated with I equivalent of alkali, in presence of phenolphthalein. From glacial AcOH it is deposited in crystals containing 1 AcOH, m. 123-4°. This comp. is not decomp, by recrystallization from ligroin. Finely divided o-benzoquinone, when treated with HCl, in Et<sub>2</sub>O (2%), does not give a quinhydrone, but is converted directly into a mixture of an insol., yellow polymer (C<sub>2</sub>H<sub>4</sub>O<sub>3</sub>)x, 4-chloropyrocatechol (27%) and 3-chloropyrocatechol (37%). The polymer fails to exhibit the quinone reactions. In abs. Et<sub>2</sub>O, Ag<sub>2</sub>O + Na<sub>2</sub>SO<sub>4</sub> oxidize 4-chloropyrocatechol to 4-chloro-obenzoquinone, O: C,H,Cl: O; lustrous, dark red plates from Et,O, long needles from hexane, in thin layers the color is light yellow, in greater thickness dark red. The quinone, m. about 78° (decomp.) and decomp. at the ordinary temp. after 1-2 dys.; it colors the skin dark brown and is not markedly volatil, even with Et.O vapor. In conc. H<sub>2</sub>SO<sub>4</sub> the color is green. 3-Chioro-o-benzoquinone, is prepared in a similar manner to the preceding comp. and also by means of PbO, petroleum ether and a little Et<sub>2</sub>O; prisms which are light yellow to bright red depending on their thickness the color changes 63°, and the comp. decomp. without m. about 68°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is violet, changing to black. 4,5-Dichloro-o-benzoquinone, O: C,H,Cl2: O, is obtained from 4,5-dichloropyrocatechol' and Ag.O; silky lustrous, light yellow to yellowish red prisms and flat tablets, m. 94° (decomp.). It gives the quinone reactions and is relatively stable but when titrated with H.SO, concordant results are

obtained only with recently prepared materials. With pyrocatechol in  $C_0H_0$  but not in Et<sub>2</sub>O, it gives a quinhydrol,  $C_{12}H_0O_4Cl_4$ ; black prisms with a metallic luster, decomp. 85°. It is rather unstable and easily evolves HCl. In  $C_0H_0$ , or  $H_2O$  the color is greenish yellow, in conc.  $H_2SO_4$  green. The monochloro-o-benzoquinones and those free from halogen do not form quinhydrols. The light color of the 4,5-dichloro-o-benzoquinone crystals may be due to the presence of a colorless peroxide (see preceding abstr.).

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α,β-Dimethyllevulic Acid. RICHARD WILLSTÄTTER AND ALESSANDRO BROSSA. Chem. Lab. Federal Techn. Hochsch., Zurich. Ber., 44, 2191-4.—Dimethyl-a, \(\beta\)-dimethylsuccinate, MeO,CCMeAcCHMeCO,Me, is obtained from Me a-bromopropionate, Me methylacetoacetate and MeONa; do 1.157. Yield, poor. By the use of EtONa a mixture of the di-Me and di-Et esters is formed. Yield, 33%. B. with Ba (OH), (2 pts.), as 8% aq. soln., hydrolyzes the ester to a mixture of dimethylsuccinic and dimethyllevulic acids. A large portion of the former is deposited from CHCl, by prolonged standing, at the ordinary temp., the remainder is separated by slow distillation under reduced pressure. α,β-Dimethyllevulic acid, AcCHMeCHMeCO,H, is more volatil than the dimethylsuccinic acid; colorless, viscid oil with a strongly acid reaction, b, 120°; b, 145°; d4 1.112. It was probably not completely purified. Ethyl ester, from alc. HCl (5%), at the ordinary temp.; mobil liquid, b, 90°; d<sub>4</sub>0.999. Barium salt, syrup. Semicarbazone and acetyl derivative, oily. p-Nitrophenylhydrazone, yellow, rounded, rhombic plates from toluene, m. 121-3° (decomp.), previously softening. Anhydride (dimethylangelica lactone), C,H1,0O,, by distilling the acid, or by heating it with mineral acids; mobil, neutral liquid, b. about 16 114°; b<sub>727</sub> 235-7°; d<sub>4</sub>0 1.08 4. It reacts with "permanganate," but does not decolorize Br, in CHCl, or in CS, I. BISHOP TINGLE.

Preparation of Methylene Derivatives. H. STAUDINGER AND O. KUPPER. Chem. Inst. Techn. Hochsch., Karlsruhe. Ber., 44, 2194-7.—A number of expts, have been made with the object of isolating methylene derivatives, CRR', in which the C at. is linked to 2 other similar, or dissimilar ats. The vapor of various substances, u der 15-20 mm., was passed over an electrically heated, glowing Pt spiral and then rapidly cooled to —20°, or to —80°. The products were identical with those obtained by others and methylene derivatives could not be isolated. Contrary to Nef's assumption, the formation of benzoin is not a methylene reaction, as will be shown later. The primary product of the decomp. of benzilic acid is diphenyl ketene (see following abstr.) and not diphenylmethylene, as stated by Nef.

Action of Hydrazine on Carbonyl Compounds. H. STAUDINGER AND O. KUPFER. Chem. Inst. Techn. Hochsch., Karlsruhe. Ber., 44, 2197-212; see preceding abstr.—Benzilhydrazone when heated during 2 hrs., at 240° (14 mm.), gives chiefly bisbenzil-ketazine, together with a little desoxybenzoin. Benzaldiphenylenemethyleneketazine, C<sub>30</sub>H<sub>14</sub>N<sub>2</sub>, is obtained by b. BzH and fluorenonehydrazone with alc.; crystals from alc., m. 82-4°. Fluorenoneketazine is formed from fluorenonehydrazine, at 200° (18 mm.), during 4 hrs. At 200°, during 6 hrs., N<sub>2</sub>H<sub>4</sub>.H<sub>2</sub>O converts it into fluorene, which is also produced from fluorenone and N<sub>2</sub>H<sub>4</sub> (3 pts.), at 200°. Diphenylene-azomethylene formula (I) below, is prepared by shaking fluorenonehydrazone with C<sub>2</sub>H<sub>4</sub>

C<sub>2</sub>H<sub>4</sub> C N (II) (II) R 
$$\stackrel{C_4H_4}{\sim}$$
 (III)

and yellow HgO, during 48 hrs.; well developed, deep red needles from petroleum ether, m. 94-5°, decomp. 159-60°; mol. wt., in freezing C<sub>6</sub>H<sub>6</sub>, 182. It is converted into dibiphenyleneethylene by the following reagents: (a) C<sub>6</sub>H<sub>6</sub> + CO<sub>5</sub>, at 130-40°;

(b) I + alc., either when heated, or at the ordinary temp.; (c) dry HBr, in b. xylene; (d) b. during 2 hrs. with alc. (NH<sub>4</sub>)<sub>8</sub>S. B., aq. NaOH + Zn dust reduce (I) to fluorene. Dimethoxybenzophenonehydrazone, C114H110-N21 is prepared from the ketone and N2H41 in alc., at 170°; broad plates from alc., m. 84-6°. Dimethoxydiphenylbenzalketazine, C<sub>2</sub>H<sub>20</sub>O<sub>2</sub>N<sub>21</sub> from the preceding compd. and BzH, in alc.; yellow crystals from alc., m. 173-4°. Dimethoxybenzophenoneketazine, C20H24O4N2, is formed by heating the preceding hydrazone at 280° (20 mm.), during 3 hrs., or by its interaction with I, in alc.; light violet needles from alc., m. 124-5°. At 200°, with N<sub>2</sub>H<sub>4</sub> in excess, it is reduced to dimethoxydiphenylmethane. Dimethoxydiphenylazomethylene (II), is prepared from the hydrazone and yellow HgO, in the manner described for (I), the reaction being continued during 24 hrs.; small, deep violet, rhombic crystals from petroleum ether, m. 103-4°; mol. wt., in freezing C<sub>6</sub>H<sub>6</sub>, 240. The comp. is unstable and in air is changed quickly to the ketazine; at 150°, in C<sub>2</sub>H<sub>4</sub> + CO<sub>2</sub>, during 6 hrs., it forms tetramethoxytetraphenylethylene. Prolonged shaking with O, in C.H. regenerates dimethoxybenzophenone from (II). Tetramethyldiaminobenzophenonehydrazone is converted into the ketazine after shaking, during 8 dys., with C.H. and yellow HgO. No azo derivative could be obtained. At 200°, during 14 hrs., excess of N<sub>2</sub>H<sub>4</sub> reduces the ketazine to tetramethyldiaminodiphenylmethane. The hydrazone is converted into the ketazine at 200° (14 mm.), during 2 hrs. At 200°, during 12 hrs., benzophenone is reduced completely to diphenylmethane by the action of N<sub>\*</sub>H<sub>\*</sub> (2.5 pts.). The same result is obtained, under similar conditions, with benzophenonehydrazone, or benzophenoneketazine and N<sub>2</sub>H<sub>4</sub> (0.5 pt.), in C<sub>2</sub>H<sub>4</sub>, but with C<sub>2</sub>H<sub>4</sub> alone, at 200°, the hydrazone is not changed. At 150°, under pressure, diphenylazomethylene is transformed completely into the ketazine. The ketazine is not decomp. after 16 hrs., at 200°. No change was produced in diphenylazomethylene by shaking with C<sub>8</sub>H<sub>e</sub> +O, during many ds. At 200°, during 14 hrs. benzalazine is reduced completely to toluene by N<sub>2</sub>H<sub>4</sub> (2.5 pts.). The same result is obtained from BzH. Stilbene is not an intermediate product in these reactions, because it is not produced from benzalazine during 14 hrs., at 200° and under these same conditions, stilbene and N<sub>2</sub>H<sub>4</sub> fail to react. These results are discussed at great length and the conclusion is drawn that the compds. of  $N_0H_A$  with "ordinary" aidehydes, ketones, esters of  $\alpha$ -ketonic acids and benzil have an identical structure. This may be either the usual hydrazone formula, RR'C: NNH, or (III) (R and R' may be either alike, or different). It is, at present, impossible to decide between these formulas. The various N<sub>2</sub>H<sub>4</sub> compds. of the ketones and aldehydes mentioned above, exhibit, in their reactions, only a difference of degree and not of kind. These dissimilarities are, of course, regarded as resulting from the varying influence of R and R'. [The authors fail to state which of the substituted benzophenones they employed in these expts. ABSTR.]

Benzylindene. R. Weissgerber. Ber., 44, 2216; see C. A., 5, 3064.—The benzylindene (m. 33-4°) described previously, is identical with the liquid compd. (b<sub>11</sub> about 180°) which J. Thiele isolated (Ann., 347, 250 (1906)) from the mixture prepared by W. Marckwald's method (Ber., 33, 1505 (1900)). J. BISHOP TINGLE.

Tetra- and Pentamethylorcinol. J. HERZIG AND F. WENZEL. I. Chem. Lab. Imp. and Roy., Univ. Vienna. *Monaish.*, 32, 461-89.—Tetra- and *pentamethylorciniol*, formula (I) below, are obtained by fractionating the product from the inter-

action c. orcinol, EtONa and MeI. The portion b<sub>19</sub> up to 140° is heated with aq. HI (d. 1.7) until the formation of MeI ceases. That part of the resulting material

which is insol. in aq. KOH is distilled and the fraction  $b_{14}$  118-24° is separated into tetramethylorcinol and (I) by freezing; cryst., m. 8°;  $b_{13}$  120°. Total yield, of both compds., 10%. Dibromotetramethylorcinol (II), is prepared in CCl<sub>4</sub>; crystals of the monoclinic system from petroleum ether, a:b:c 0.724 3:1:0.9614; ac 96° 18′; m. 79.5°. Yield, almost quant. Aq. KOH (0.4%) converts it into a mixture of fumaric acid (12.4%) diisopropyl ketone (10.7%) and an acid,  $C_{11}H_{14}O_4$ ; crystals from  $H_2O$ , m. 165°. Yield, 66.5%. Methyl ester,  $C_{10}H_{14}O_3CO_4Me$ , from the acid and diazomethane, is purified by means of alc.  $+H_2O$ , m. 64-7°. Bromopentamethylorcinol (III), is prepared in a similar manner to (II) and is purified by distillation (17 mm.); tabular crystals of the monoclinic system from petroleum ether, a:b:c 1.4653: 1:0.7528; ac 91° 3′; m. 43-5°;  $b_{14}$  117°; mol. wt., in PhNH<sub>2</sub>, (Bleier-Kohn) 212. Yield, 73%. The reactions described above are discussed at great length, but no definit conclusions are reached.

Alkylation in the Nucleus. J. HERZIG AND BR. ERTHAL. I. Chem. Lab. Imp. and Roy., Univ. Vienna. Monatsh., 32, 491-504.—Unless otherwise stated the expts. described below were carried out by means of alkyl iodides and aq. KOH (10%). Tetramethylphloroglucinol gave a mixture of penta- and hexa-Me derivatives. Tetraethylphloroglucinol gave the methoxy compd., which is not hydrolyzed by alc. KOH. The tri-Me ether was the chief product from phloroglucinyl dimethyl ether and MeI. Orcinol gave a mixture of tetra- and penta-Me derivatives. Tetramethylorcinol remained unchanged by further treatment with MeI and KOH soln. (20%). Resorcinol gave trimethylresorcinyl methyl ether (20% KOH). Hydroquinyl dimethyl ether was obtained from hydroquinol. Phloroglucinol, EtI and aq. KOH (20%) gave pentaethylphloroglucinyl ethyl ether. From Ag phloroglucinylcarboxylate and Etl, Et phloroglucinylcarboxylate was produced. Yield, 56%. Orcinyl dimethyl ether is obtained from orcinol and diazomethane. Phloroglucinol and Me SO, give phloroglucinyl trimethyl ether. Orcinol, under similar conditions, forms orcinyl dimethyl ether. Diazomethane and succinylosuccinic ester do not react, but di-Et phloroglucinyldicarboxylate, under similar conditions, forms the tri-Me ether, (MeO)<sub>2</sub>C<sub>2</sub>H(CO<sub>2</sub>Et)<sub>2</sub>. Yield, good. The results described above are discussed at considerable length, but no definit conclusions are reached. J. BISHOP TINGLE.

Hexa- and Pentamethylphloroglucinol. J. Herzig and Br. Erthal. I. Chem. Lab. Imp. and Roy., Univ. Vienna. Monatsh., 32, 505-8.—Hexamethylphloroglucinol fails to react with NH<sub>2</sub>OH, PhN<sub>2</sub>H<sub>3</sub>, p-nitrophenylhydrazine, semicarbazine, or amyl nitrite and it is not reduced in presence of Pt. With MeMgI it gives a compd., possibly 1,3,5-trihydroxynonomethylhexahydrobenzene, (HO)<sub>2</sub>C<sub>2</sub>Me<sub>3</sub>; crystals from acetone, m. 258-60°. Yield, 80% of the phloroglucinol. With a large variety of reagents which were tried the compd. either failed to react, or decomp. completely. Pentamethylphloroglucinyl methyl ether and MeMgI give what is probably 1,3,5-trihydroxyheptamethyltetrahydrobenzene, (HO)<sub>2</sub>C<sub>3</sub>Me<sub>7</sub>; crystals from petroleum ether, m. 67-8°.

J. BISHOP TINGLE.

Exchange of Primary, Secondary and Tertiary Alkyls in the Case of Esters of Organic Acids. MICHAEL PFANNL. II. Chem. Lab., Univ. Vienna. Monatsh., 32, 509–22; see C. A., 5, 1087 and T. Komnenos, C. A., 5, 1745, 1919.—BzOMe (1 mol.) trimethylcarbinol (10 pts.) and K trimethylcarbinolate (1 mol.), when heated, give, chiefly, tert.-butyl benzoate, Me<sub>2</sub>COBz; highly refractive, oily liquid with the characteristic odor of benzoic esters, b<sub>10</sub> 94°, b. 213°. Under the ordinary pressure it decomp. slowly at the b. p. into BzOH and isobutylene, Me<sub>2</sub>C: CH<sub>2</sub>. Only about 20% of the K trimethylcarbinolate employed reacted so as to cause hydrolysis. BzOMe, b<sub>10</sub> 75–6°. Under similar conditions, after 24 hrs., at the ordinary temp., di-Me tere-

phthalate (1 mol.) butyl alc. and K butylate (0.1 mol.) give dibutyl terephthalate; crystallin, m. 16°. Yield, quant. A similar expt. with isobutyl alc. and K isobutylate (0.05 mol.) gave diisobutyl terephthalate, m. 55°. Yield, 95.4% of the di-Me ester. The quantity of ester hydrolyzed was only equal to about 30% of the K alcoholate. In the case of Pr<sup>B</sup>OH the reaction was stopped after 30 mins.; 0.5 mol. of Pr<sup>B</sup>OK was employed. The yield of diisopropyl terephthalate was 70% of the di-Me ester. Isopropyl terephthalate, Pr<sup>B</sup>O<sub>2</sub>CC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H, is formed together with the diester; slender needles from benzine, m. 166°. Sodium salt, m. 166°. Di-Me terephthalate and K tert.-butylate, in quantity insufficient to form the di-tert.-butyl ester, give only di-K terephthalate and unchanged di-Me ester. The results were the same either at the ordinary temp., or at the b. p. With 0.5 mol. of K tert.-butylate, b. during 1.5 hrs., 50% of the di-Me ester was hydrolyzed and the remainder converted into equal proportions of di-tert.-butyl and Me tert.-butyl terephthalates. The method described above is well suited for the preparation of esters of organic acids, in cases where esters derived from the lower homologous alcs. are more easily obtained than those derived from alcs. higher in the series. J. BISHOP TINGLE.

Electrolysis of Salts of Aliphatic Acids in Solutions of their Respective Anhydrous Parent Acids. K. HOPFGARTNER. Chem. Lab. Imp. and Roy., Univ. Innsbruck. Monatsh., 32, 523-61.—The gases produced by the electrolysis of anhydrous AcONa, in glacial AcOH, consist of CO<sub>2</sub> (49.28), C<sub>2</sub>H<sub>4</sub> (1.17), O (0.16), C<sub>2</sub>H<sub>4</sub> (21.06) and H (27.85) vol. %. Pt electrodes were employed. The use of glacial AcOH containing 10% (by vol.) of Ac<sub>2</sub>O produced no marked effect on the nature and proportion of the gaseous products. A large number of expts. were made in order to determin the influence of conc., temp. and current density on the electrolysis of AcOK, in AcOH. In no case was there attained the vol. ratio  $CO_2 : C_2H_6 : H = 2 : 1 : 1$ . The nearest approach to this was 2.032: 0.993: 1, together with 0.010 of C.H. and 0.006 of O. These results were obtained with the conc. 8.8%, at 14-15°, with a current density of 12.8 amperes per sq. dm. The lowest result was  $CO_2 : C_2H_4 : H : C_2H_4 : O = 1.595 :$ 0.751: 1: 0.015: 0.008, conc. 17.4%, at 43-8°, current density 0.38 ampere per sq. dm. The results with Na propionate, in propionic acid, are illustrated by the values CO<sub>2</sub> (45.14), C<sub>2</sub>H<sub>4</sub> (9.01), O (0.28), C<sub>4</sub>H<sub>10</sub> (12.13), H (33.89) vol. %. Similar representative results, obtained with HCO<sub>2</sub>Na, in HCO<sub>2</sub>H, are CO<sub>2</sub> (44.36), O (0.52), CO (0.66) and H (54.43) vol. %. In general, the author's expts. show that the electrolysis of alkali salts of the acids mentioned, in the resp. acids, gives products which are qualitatively the same as those produced by the use of the salts in acidified aq. solns. The ratio of the products is, however, often very different in the 2 cases. The exceptions are the formation of CO and probably of butylene, from HCO, Na and from Na propionate, resp. These do not appear to be produced in aq. soln. The influence of increase in temp., conc. and current density differs in degree, but not in kind, in the case of the aq. and acid solns. A variation in the current density has but very little effect on the electrolysis in the solns. in acids. The reactions which actually take place in the solns. of acids must be formulated in such a manner as to exclude H<sub>2</sub>O mols. and probably the same should apply to the reactions in H<sub>2</sub>O solns. J. B. T.

Chemical Action of Light. XIX, XX, XXI. G. CIAMICIAN AND P. SILBER. Atti accad. Lincei, 20, I, 714-21, 721-4, 881-5; see C. A., 5, 3238.

J. B. T.

Oxyazo Compounds and Ketohydrazones. V. Phenylhydrazone Derivatives of Thionaphthenequinone. K. Auwers and K. Müller. Chem. Inst., Univ. Greifswald. Ann., 381, 276–303; see C. A., 5, 3416.—Thionaphthene-β-phenylhydrazone formula (I) below, does not react with b. Ac<sub>2</sub>O, nor with PhN<sub>2</sub>H<sub>3</sub>, up to 140°. Alc. alkali hydrolyzes it to o-thiophenylglyoxylic acid phenylhydrazone, HSC<sub>2</sub>H<sub>4</sub>C(: NNHPh)-CO<sub>2</sub>H; unstable, amorphous powder, m. 80–6°. Warm alc., or glacial AcOH recon-

verts it into (I). Methyl o-methylthiophenylglyoxalate phenylhydrazone, MeSC<sub>2</sub>H<sub>4</sub>C-(: NNHPh)CO<sub>2</sub>Me, is obtained from the preceding compd., MeI and MeONa, in

$$C_{e}H_{e} < C(:NNHPh) > CO$$

$$C_{e}H_{e} < C(:NNPhBz) > CO$$

$$C_{e}H_{e} < C(:NNPhBz) > CO$$

MeOH; flat, pale yellow needles from alc., or ligroin, m. 107.5°. It is hydrolyzed by alc. NaOH to o-methylthiophenylglyoxalic acid phenylhydrazone, MeSC<sub>e</sub>H<sub>4</sub>C(: NNHPh)-CO<sub>2</sub>H; small, S-yellow crystals from alc., m. 180° (decomp.). Thionaphthenequinone-β-benzoylphenylhydrazone (II), is prepared from thionaphthenequinone and asym. benzoylphenylhydrazine hydrochloride, in hot alc.; flat, lustrous, deep red needles, m. 199°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is brown. It is very readily hydrolyzed to (I) by almost all reagents, including alc. at 150°. In AcOEt, Zn dust and glacial AcOH reduce it to benzanilide, no trace of PhNH<sub>2</sub> being formed. Thionaphthenequinone-β-methylphenylhydrazone (III), is obtained by b. the quinone with asym. methylphenylhydrazine,

$$C_{\bullet}H \stackrel{C(: NNMePh)}{\underbrace{\qquad}} CO \qquad C_{\bullet}H \stackrel{CO}{\underbrace{\qquad}} C: NNHPh$$

or its hydrochloride and alc., or glacial AcOH; dark red needles with a dull luster from alc., m. 98-9°. In conc. H<sub>2</sub>SO<sub>4</sub> it gives a deep, pure mallow color. Alc. NaOH hydrolyzes it to a yellow, amorphous, unstable acid. At 140°, excess of asym. methylphenylhydrazine converts (III) into a compound, probably a stereoisomer; small, opaque, S-yellow crystals from MeOH, m. 122-3°. Like (III) it does not give even a trace of PhNH<sub>2</sub> when reduced by means of Zn and AcOH. Thionaphthenequinone-a-phenylhydrazone (IV) gives a cherry-red color in conc. H<sub>2</sub>SO<sub>4</sub>. It was prepared from phenyldiazonium chloride and hydroxythionaphthene, or its carboxylic acid and also from PhN<sub>2</sub>H<sub>2</sub> and 2-dibromo-3-ketodihydrothionaphthene, perferably in cold CHCl<sub>2</sub>. At a high temp., more slowly at 100°, PhN<sub>2</sub>H<sub>2</sub> and (IV) give thionaphthenequinoneosazone (V); thin, orange-yellow needles from C<sub>6</sub>H<sub>6</sub>, m. 199-200°. It is

decomp. by prolonged heating with glacial AcOH; in conc. HCl the color is brownish violet. Thionaphthenequinone-α-benzoylphenylhydrazone (VI), is prepared from (IV), BzCl, aq. NaOH (9%) and alc., at the ordinary temp., or in presence of pyridine, provided that the BzCl is in large excess (5 mols.); in this last case the product is orangecolored, m. 136-40°. When once recrystallized from alc. ruby-red, rhombic prisms, or carmin-red prisms are deposited, m. 141°. Its color in neutral solvents is orangeyellow, in conc. H<sub>2</sub>SO<sub>4</sub> cherry-red. Further recrystallization from alc., or glacial AcOH converts the red compd. into opaque, lustrous, orange-yellow prisms, m. 156°. The compds. have the same composition, each is hydrolyzed to (IV) by alc. alkali hydroxides and, in common with the original crude material, each modification is reduced by Zn dust and glacial AcOH, at the ordinary temp., or at -5°, to benzanilide, no trace of PhNH, being formed. Thionaphthenequinone-a-methylphenylhydrazone (VII), is prepared from (IV) and Me<sub>2</sub>SO<sub>4</sub>, or by b. (IV) with MeI, MeONa and MeOH and is separated from (VIII) or (IX) by its insolubility in cold Et<sub>2</sub>O; long, red, glassy lustrous, flat needles from alc., m. 133-4°. Its color in conc. H<sub>2</sub>SO<sub>4</sub> is brownish orangeyellow. It gives MeNHPh, but no PhNH, when reduced by means of Zn dust and glacial AcOH. At the ordinary temp., in CHCla, asym. methylphenylhydrazine and

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2-dibromo-3-ketodihydrothionaphthalene combine to form (VII). α-Phenylazo-β-

$$C_0H_{\bullet} \subset CO \subset C$$
: NNMePh  $C_0H_{\bullet} \subset COMe$   $COMe$   $COM$ 

methoxythionaphthene (VIII), or the C-Me compd. (IX), is formed together with (VII); aggregates of orange-yellow plates from MeOH, m. 133-4°. A mixture of it with (VII) m. 110°. Its solns. in ordinary media are golden yellow, in conc. H<sub>2</sub>SO<sub>4</sub> brownish red. When reduced by means of Zn dust and glacial AcOH it gives PhNH<sub>2</sub>, but it is, at present, impossible to decide if it be represented by (VIII) or (IX). It could not be

transformed into (VII) nor could the change of this into (VIII), or (IX) be effected. Benzoyl-α-thionaphthenequinoneoxime (X), is best prepared from the oxime, in presence of pyridine; S-yellow, lustrous plates from C<sub>6</sub>H<sub>6</sub>, giving a greenish yellow powder, m. 170°. Aq. NaOH hydrolyzes it to thiosalicylic acid. With PhN<sub>2</sub>H<sub>2</sub>, in hot alc. (X), gives (IV). Thionaphthenequinone-α-oxime-β-phenylhydrazone (XI), was prepared

$$C_4H_4$$
 $C(: NNHPh)$ 
 $C: NOH$ 
 $C_4H_4$ 
 $C(: NNHPh)$ 
 $C: NOAc$ 

from thionaphthenequinoneoxime and PhN<sub>2</sub>H<sub>3</sub>, in glacial AcOH, on the H<sub>2</sub>O bath; silky lustrous, golden yellow, interlaced needles from alc., m. 172°. When mixed with the oxime, m. 134-50°. The compd. does not appear to be identical with the substance of the same name described in Ger. Pat. 213,458. B. PhN<sub>2</sub>H<sub>3</sub> quickly converts (XI) into (V), but at the H<sub>2</sub>O bath temp. no change is produced. Acetyl derivative (XII), is formed from (XI) and AcCl, in pyridine; opaque, lustrous, golden yellow prisms rom C<sub>2</sub>H<sub>6</sub>, m. 156-7°. Benzoyl derivative is obtained in a similar manner; golden yellow, interlaced needles from ligroin, m. about 160°. It could not be brought to a constant m. p. Thionaphthenephenylosotriazole (XIII), is formed by treating

(XII), or the preceding compd. with alc.- $H_2O$  NaOH, or by heating with glacial AcOH; silky lustrous, colorless needles, m.  $152^{\circ}$ . It is very stable and gives no color with conc.  $H_2SO_4$ . 4-Methylthionaphthenequinone- $\alpha$ -phenylhydrazone (XIV), is prepared from phenyldiazonium chloride and 4-methoxythionaphthene; small, flat, dark brick-red needles with a strong greenish yellow surface luster from glacial AcOH +  $H_2O$ , m.  $186.5^{\circ}$ . In conc.  $H_2SO_4$  the color is cherry-red. 4-Methylthionaphthenequinone- $\alpha$ -benzoylphenylhydrazone (XV) is obtained from (XIV) either by the Schotten-

$$MeC_{e}H_{s} < CO > C : NNPhBz$$
 $MeC_{e}H_{s} < CO > C : NOH$ 
 $S = CO > C : NOH$ 
 $S = CO > C : NOH$ 

Baumann method, or by means of pyridine; it exists in 2 forms as does (VI). (A) Long, lustrous, orange yellow needles, m. 145°, are isolated by extracting the crude product with a little b. alc. and then recrystallizing the residue from MeOH, or alc.

The second modification (B) is produced by direct recrystallization of the crude product from  $C_0H_0$  + petroleum ether; opaque, S-yellow crystals, m. 157°. By the pyridine method of acylation (A) is formed in the larger quantity. In ordinary solvents the color of (B) is pure yellow, that of (A) orange-red; the colors in conc.  $H_2SO_4$  are brown and cherry-red, resp. Cold, aq. alkali hydroxides appear to hydrolyze (B) more rapidly than they do (A). At the ordinary temp., neither (A) nor (B) gives PhNH<sub>2</sub> by the action of Zn and glacial AcOH, but, owing to hydrolysis, some PhNH<sub>3</sub> is formed if the reducing mixture be b. during a considerable period. 4-Methylthionaphthene-quinone-a-oxime (XVI), is prepared from the quinone, NaNO<sub>2</sub> and cold, dil. HCl; slender, flat, lustrous yellow needles and plates from alc., darkens about 165° when slowly heated, heated with medium rapidity it softens about 180°, m. about 188° (decomp.). Dibenzoyl-4-methyl-1-amino-2-hydroxythionaphthene (XVII), is prepared

$$\begin{array}{c} \text{MeC}_{\bullet}\text{H}_{\bullet} & \begin{array}{c} C(OBz) \\ S \\ \hline \end{array} \\ \text{(XVII)} \end{array} \\ \begin{array}{c} C_{\bullet}\text{H}_{\bullet} & \begin{array}{c} C(OH) \\ S \\ \hline \end{array} \\ \text{(XVIII)} \end{array} \\ \end{array} \\ \text{NPh}$$

by warming (XVI) with aq. NaOH (8%) and Zn dust and adding BzCl to the mixture; white, rough plates from alc., or C<sub>6</sub>H<sub>6</sub>, m. 233.5°. The reactions described above show that the compds. have the formulas given and that they are not azo derivatives such as (XVIII). The 2 forms of (VI) and (XV) are regarded as being stereoisomers; the authors appear to think that this "explains" the difference in color shown by the members of the resp. pairs of compds. (see following abstr.). J. BISHOP TINGLE.

Oxyazo Compounds and Ketohydrazones. VI. Isatinphenylhydrazones. K. Auwers and A. Boennecke. Chem. Inst., Univ. Greifswald. Ann., 381, 303-12; see preceding abstr.—β-Isatinbenzoylphenylhydrazone formula (I) below, is obtained

by digesting isatin and asym. benzoylphenylhydrazine in highly dil. alc. soln., on a  $H_2O$  bath; slender, orange-colored needles from  $C_0H_6$ , m. 195-6°. It is hydrolyzed, at the ordinary temp., by alc. NaOH to BzOH and  $\beta$ -isatinphenylhydrazone (II). N-Benzoylisatin- $\beta$ -phenylhydrazone (III), is prepared by any of the following methods.

$$C_{\bullet}H_{\bullet} \stackrel{C(:NNHPh)}{\underset{NBz}{\longleftarrow}} CO$$
  $C_{\bullet}H_{\bullet} \stackrel{C(:NNAcPh)}{\underset{(IV)}{\longleftarrow}} CO$ 

(a) From (II), acetone, aq. NaOH and BzCl. (b) In a similar manner to (a) by the use of pyridine. (c) By b. N-benzoylisatin, in conc. alc. soln., with PhN<sub>\*</sub>H<sub>1</sub>.HCl; slender, golden yellow, lustrous, interlaced needles from glacial AcOH, m. 203°. It is hydrolyzed as readily as is (I). When reduced by means of alc., AcOH, AcOEt and Zn dust it gives PhNH<sub>2</sub>, but no benzanilide.  $\beta$ -Isatinacetylphenylhydrazone (IV), is prepared from isatin and asym. acetylphenylhydrazine in alc. and is purified by extraction with Et<sub>2</sub>O; small, octahedral, bright greenish pale yellow crystals, m. 199°. B. alc. hydrolyzes it to (II); it gives PhNHAc when reduced by means of Zn dust glacial AcOH and AcOEt. N-Acetylisatin- $\beta$ -phenylhydrazone (V), is not an O-acetyl

$$C_{\bullet}H$$
 $C(:NNHPh)$ 
 $COAc$ 
 $NAc$ 
 $(VI)$ 
 $C_{\bullet}H$ 
 $C(:NNHPh)$ 
 $COAc$ 

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derivative (VI) as stated by Schunck and Marchlewski (Ber., 28, 543), because it is formed from Ac<sub>2</sub>O and (II) and also from N-acetylisatin and PhN<sub>2</sub>H<sub>2</sub>.HCl, in alc. It is reduced, in the manner described above, to PhNH<sub>2</sub> and not to PhNHAc. N-Benzoylisatin-α-phenylhydrazone (VII), is prepared from isatin-α-phenylhydrazone,

$$C_{\bullet}H_{\bullet} \stackrel{CO}{\swarrow} C: NNHPh$$
 $C_{\bullet}H_{\bullet} \stackrel{C(:NNRPh)}{\swarrow} COH$ 
(VII)

BzCl and aq. NaOH; slender, interlaced, yellow needles from  $C_0H_6$ , m. 188-9°. It is hydrolyzed quickly by warm, alc. alkali hydroxides to isatin- $\alpha$ -phenylhydrazone and is reduced to PhNH<sub>2</sub> by Zn dust and glacial AcOH. Although their reactions and methods of formation establish the true hydrazone nature of the compds. described above, there is, at present, no means of distinguishing between (I), (II) and (IV), or of such a formula as (VIII) for the 3 compds. in question (R = H, Bz or Ac, resp.).

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Thianthrene. K. FRIES AND W. VOGT. Chem. Inst., Univ. Marburg. Ann., 381, 312-37.—Thianthrene is best prepared by adding finely divided AlCl<sub>2</sub> (100 g.) to C<sub>6</sub>H<sub>6</sub> (500 g.), cooling to 0° and then running in SCl<sub>2</sub> (200 g.) in C<sub>6</sub>H<sub>6</sub> (300 g.); after 5 hrs., at the ordinary temp., the mixture is b. with stirring and exclusion of H<sub>2</sub>O, for 2-3 hrs. and then allowed to remain 15 hrs. Yield of purified thianthrene, 136%. Ferric chloride, C<sub>12</sub>H<sub>2</sub>S<sub>2</sub>. FeCl<sub>2</sub>, is prepared in glacial AcOH, from anhydrous FeCl<sub>2</sub>; opaque needles with a bronze luster giving a violet powder. Thianthrene sulfoxide, formula (I) is prepared by adding HNO<sub>2</sub> (d. 1.2) to thianthrene, in glacial AcOH (30 pts.); m.

$$C_eH_e \stackrel{SO}{\underset{S}{\longrightarrow}} C_eH_e$$
  $C_eH_e \stackrel{SO}{\underset{SO}{\longrightarrow}} C_eH_e$   $C_eH_e \stackrel{SO_2}{\underset{S}{\longrightarrow}} C_eH_e$ 

143°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is brownish red, changing to blue with a little H<sub>2</sub>O and disappearing with more H<sub>2</sub>O. In conc. HCl (I) dissolves with relative ease, the soln. is first pale rose-colored, then deep blue and the liquid deposits chlorothianthrene gradually. The chloro compd. is also formed by dissolving (I) in glacial AcOH and satg. the soln. with HCl, (deep red at first). When Cl is passed into solns. of (I) a yellow color is developed and on shaking with H<sub>2</sub>O an excellent yield of the thianthrene disulfoxide (II) is obtained. Thianthrenesulfone (III), is prepared from Cl and thianthrene in hot, aq. AcOH, when red crystals of the dichloride begin to deposit the Cl is stopped and the liquid is b. until the red color disappears. Cl is again added and the process repeated until the cool liquid ceases to deposit red crystals. Colorless, lustrous plates from glacial AcOH, m. 159°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is pale rose; the compd. is not changed by HBr, nor by b. glacial AcOH and Zn dust, but HNO<sub>3</sub> (d. 1.4) oxidizes it to (IV) and H<sub>2</sub>O<sub>3</sub>, in glacial AcOH, converts it into the disulfone. Thianthrenesulfone

sulfoxide (IV) is best prepared from (III), but is also formed from (I) and the 2 isomeric forms of (II) by b. with HNO<sub>2</sub> (d. 1.4); small, opaque, prismatic crystals from alc., m. 216°. It gives no color with conc.  $H_2SO_4$ ; aq. HBr reduces it to (III) and CrO<sub>2</sub> oxidizes it to the disulfone. Thianthrene dichloride (V) or (VI), is always formed, even with excess of the gas, when dry Cl is passed into a soln. of thianthrene, preferably in  $C_0H_2$ ; red, unstable, prismatic crystals resembling  $K_2Cr_2O_7$ . Dry solvents decomp. it into Cl and thianthrene; with  $H_2O$  it gives HCl and (I). In aq. HCl the color is deep blue (see

(I) above). 4-Chlorothianthrene (VII), is prepared by saturating with dry HCl a soln. of (I) in glacial AcOH (5 pts.), or by adding the calculated quantity of Cl to thianthrene, in CHCl<sub>2</sub> (5 pts.); white prisms from glacial AcOH, m. 84°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is violet-rose, changing slowly at the ordinary temp., more quickly when heated, to deep violet-blue. 4,4'-Dichlorothianthrene (VIII) is prepared by adding the calculated quantity of Cl to thianthrene, or to (VII), in CHCl<sub>2</sub>; from (I) and HCl, in glacial AcOH and

$$CI \longrightarrow S \longrightarrow C_0H_4 \qquad CIC_0H_3 \bigcirc C_0H_3CI \qquad \underbrace{\begin{pmatrix} OEt \\ \frac{3}{2} & 1 \\ \frac{5}{6} & 6 \end{pmatrix}}_{OEt} \longrightarrow CO_2 \longrightarrow \underbrace{\begin{pmatrix} SO_2H \\ \frac{2'}{3} & 3' \\ \frac{6'}{5} & 4' \end{pmatrix}}_{OE}$$

from PhCl, SCl<sub>2</sub> and AlCl<sub>2</sub>, in CS<sub>2</sub>, first in a freezing mixture then at the b. p. during 20 hrs.; long, slender needles from glacial AcOH, or C<sub>6</sub>H<sub>6</sub>, m. 171°. Yield, about 22% of the PhCl. In conc. H<sub>2</sub>SO<sub>4</sub> the color is pale bluish violet, changing to blue. 2-Ethoxydiphenylsulfone-2'-sulfinic acid (IX), is prepared by b. thianthrenedisulfone with alc. and aq. KOH (50%), the product being acidified with conc. HCl; long, slender needles from alc. + dil. HCl, m. 151° (decomp.). Yield, almost quant. Sodium salt, thin bundles of needles. It is reduced in alc., by Zn dust and HCl to 2-ethoxy-2'mercaptodiphenylsulphone, SHC<sub>2</sub>H<sub>4</sub>SO<sub>2</sub>C<sub>2</sub>H<sub>4</sub>OEt; small needles from alc., m. 131°. When warmed with conc. H<sub>2</sub>SO<sub>4</sub> a pale red color is produced. Methyl ether, C<sub>18</sub>H<sub>16</sub>O<sub>2</sub>S<sub>2</sub>, from Me.SO4 opaque needles from CaHe, m. 178°. 2-Ethoxydiphenylsulfone 2'-disulfide, (EtOC<sub>2</sub>H<sub>4</sub>SO<sub>2</sub>C<sub>2</sub>H<sub>4</sub>S)<sub>2</sub>, is prepared from (IX) and HBr in b. glacial AcOH, or by oxidizing the mercaptan by means of K, Fe(CN), or of air and alkali; small, opaque crystals from glacial AcOH, m. 270° (decomp.). In alc. alkali, Na,S regenerates the mercaptan. 2-Ethoxydiphenylsuljone-2'-suljonyl chloride, EtOC,H,SO,C,H,SO,Cl, from the sulfinic acid and Cl, in hot, glacial AcOH; opaque, rhombic crystals from C.H., m. 159°. 2-Ethoxydiphenylsulfone-2'-sulfonyl bromide is prepared in a similar manner to the preceding compd., from the calculated quantity of Br; opaque, rhombic crystals, m. 177°. With Br in excess the sulfinic acid gives a compound, C14H12OABr2S2; needles from glacial AcOH, m. 179°. It forms a perbromide, C14H12O5Br2S2.Br2; orangecolored needles from glacial AcOH. It is stable in air at the ordinary temp. 2-Ethoxydiphenylsulfone-2'-sulfonic acid, EtOC<sub>0</sub>H<sub>4</sub>SO<sub>2</sub>C<sub>0</sub>H<sub>4</sub>SO<sub>2</sub>H, is prepared by adding H<sub>2</sub>O to a b. glacial AcOH soln. of the sulfonyl chloride, or bromide; when the mixture becomes turbid it is b. until H<sub>2</sub>O produces no further effect; opaque needles containing AcOH, from glacial AcOH, after drying, m. 178°. Anilide, C20H19O5NS2, from the sulfonyl chloride or bromide; prismatic crystals from glacial AcOH, m. 204°. It dissolves in warm, aq. NaOH and is repptd. by acids. 2-Methoxydiphenylsulfone-2'sulfinic acid, MeOC.H.SO.C.H.SO.H, is prepared in a similar manner to (IX) from MeOH; long crystals, m. 161° (decomp.). Yield, almost quant. It resembles (IX) in general properties. 2-Methoxy-2-mercaptodiphenylsulfone, MeOC,H,SO,C,H,SH, long, flat prisms, or short, opaque, rhombic crystals from alc., m. 157°. Methyl ether, C14H14O2S2, from the mercaptan, Me2SO4 and alkali; opaque prisms from C6H6, m. 197° 2-Methoxydiphenylsulfone-2'-sulfonyl chloride, MeOCaHaSOaCaHaSOaCl, opaque prisms from glacial AcOH, m. 210°. 2-Methoxydiphenylsulfone-2'-sulfonic acid, MeOC<sub>0</sub>H<sub>4</sub>SO<sub>3</sub>-C.H.SO.H., opaque needles from glacial AcOH, m. 202°. Anilide, from the sulfonyl chloride; rhombic crystals from glacial AcOH, m. 193°. The 5 preceding compds. resemble the corresponding ethoxy derivatives closely and are prepared in a similar J. BISHOP TINGLE. manner to them.

Chlorides and Bromides of Diphenyl Sulfide. K. FRIES AND W. VOGT. Chem. Inst., Univ. Marburg. Ann., 381, 337-46.—At o°, dry Cl and diphenyl sulfide, in C.H., give a very unstable dichloro additive compound; pale yellow, flat, prismatic

crystals. It gradually evolves HCl and decomp. into a mixture of mono- and dichlorodiphenyl and diphenyl sulfides. In presence of H<sub>2</sub>O, the additive compd. forms HCl and diphenyl sulfoxide and it liberates I from KI. 4,4'-Dichlorodiphenyl thiodichloride, (ClC<sub>2</sub>H<sub>4</sub>)<sub>2</sub>SCl<sub>2</sub>, is prepared most easily from diphenyl sulfide and Cl<sub>2</sub> in C<sub>2</sub>H<sub>2</sub>, and is also formed from 4,4'-dichlorodiphenyl sulfide; opaque, prismatic crystals resembling K<sub>4</sub>Fe(CN)<sub>6</sub> in color, decomp. and evolves Cl about 95°, the residue consists of pure 4,4'-dichlorodiphenyl sulfide. It is fairly stable in dry air but H<sub>2</sub>O decomp. it into HCl and 4,4'-dichlorodiphenyl sulfoxide, (CIC,H<sub>4</sub>),SO, which is also prepared by b. 4,4'-dichlorodiphenyl sulfide with HNO<sub>2</sub> (d. 1.4); small, opaque crystals from benzine, m. 143% In conc. H.SO. the color is pale violet. The sulfoxide is reduced to the sulfide by the action of HBr. Diphenyl thiodibromide, C12H10Br2S, is prepared in hexane, at the temp. of NaCl + ice; orange-colored, unstable needles. Alone, or with H<sub>2</sub>O. it passes into a mixture of diphenyl sulfide and its mono- and dibromo substitution products; very little sulfoxide is produced. In CHCla. NaHSOa soln. reduces it to diphenyl sulfide, showing that the halogen is linked to the S at. In warm hexane, 4,4'-dibromodiphenyl sulfide and Br give 4,4'-dibromodisulfide thiodibromide, C12H2Br4S; dark red, lustrous, unstable leaflets. With a larger proportion of Br, as above, 4,4'dibromodiphenyl thiotetrabromide, C12H2Br6S, is produced; brownish red, opaque prisms with a bronze luster. It readily decomp. into the sulfide and Br. 4,4'Dibromodiphenylsuljoxide, (BrC<sub>2</sub>H<sub>4</sub>)<sub>2</sub>SO, is prepared from the thiodichloride, or from the sulfide and HNO<sub>2</sub> (d. 1.4); lustrous, lancet-shaped crystals from alc., m. 152°. It dissolves in conc. aq. HCl and gives a pale violet color with conc. H<sub>2</sub>SO<sub>4</sub>. In glacial AcOH, HBr reduces it to the sulfide. These results show that, contrary ro the statement of Fromm and Raiziss, tert. S (Ph.S, etc.) can form additive compds. (see C. A., 4, J. BISHOP TINGLE. 2307).

Colorimetric Dilution Law and its Application to Triphenylmethyl. JEAN PICcard. Chem. Lab. Roy. Acad. Sci., Munich. Ann., 381, 347-51.—As is well known, Beer's law states that, provided the solvent is inert, the color of the soln. of a substance is constant and independent of the dilution, if the colored material consists of 2 isomeric modifications which are in equilibrium. In case dilution causes the resolution of a polymeric system into simpler mols., the law no longer applies. If both modifications are colored, further diln. produces a change in tint and if only I form is colored diln. causes an alteration in the intensity of the color of the soln. These considerations have been applied to the study of a number of solns. by Horn and also by Hantzsch. An investigation of "triphenylmethyl," in Et,O and of "tribiphenylmethyl," shows that they undergo dissociation from the ethane complex, R<sub>2</sub>C<sub>2</sub>, to R<sub>2</sub>C, as diln. increases. The proportion of R<sub>4</sub>C which is present in any soln. depends on the conc.; if the soln. is caused to crystallize, only the polymeric form, R<sub>2</sub>C<sub>2</sub>, is deposited, because the equilibrium is disturbed. Consequently, the nature of the crystals which are obtained does not throw any light on the proportions of the 2 polymers which were present in soln. Schmidlin (C. A., 2, 2944) has ignored these conclusions.

Simpler Quinoidal Dyes. Jean Piccard. Chem. Lab. Roy. Acad. Sci., Munich-Ann., 381, 351-66.—Meriquinonediimmonium bromide,  $C_{12}H_{16}N_4Br_2$ , was prepared from p-phenylenediamine and Br, in glacial AcOH + abs. alc., at the temp. of a freezing mixture; flat, microscopic prisms with a coppery luster. It is stable in dry air, but is decomp. with great readiness by  $H_2O$ . At 0°, 0.3 g. in  $H_2O_4$ . (1) is bluish green. at 20° yellow; at 0°, more dil. solns. are yellow ( $\alpha$ -form), but change to blue ( $\beta$ -form) by adding NaCl. Meriquinonediimmonium nitrate,  $C_{12}H_{16}O_4N_6$ , is prepared from p-phenylenediamine, dil alc., HNO<sub>2</sub> and N oxides, at the temp. of ice + CaCl<sub>2</sub>; small crystals with a bronze luster. It resembles the bromide. Meri-N-methylquinonediimmonium bromide,  $C_{14}H_{20}N_4Br_2$ , is prepared in a similar manner to the preced-

ing bromide, as is also asym. meri-N-diethylquinonediimmonium bromide, which was obtained only in soln. It closely resembles the di-Me derivative. Meri-N-trimethylqui-menediimonium ferricyanide, C<sub>18</sub>H<sub>48</sub>N<sub>12</sub>Fe, from K<sub>2</sub>Fe(CN)<sub>6</sub>, N-trimethyl-p-phenylenediamine and dil. AcOH; bundles of black crystals, The above meriquinoids are formulated

X =an acid radicle). Many of these compds. exist in 2 forms, of which the  $\alpha$ - is unstable in the solid state and dissolves readily in glacial AcOH. The  $\beta$ -modification is stable and does not dissolve in glacial AcOH. A study of the absorption spectra shows that, as the number of Me groups in the mol. increases from o to 4, the color changes successively from yellow to orange, red and violet to blue. The absorption spectra of the 5 compds, indicated ( $\alpha$ -forms) are very similar in detail. The  $\beta$ -forms of meriquinonediimmonium bromide and of its Me and di-Me homologs also have spectra (blue) which are very similar in character, but they are quite different from the spectra of the  $\alpha$ -forms. The spectra are illustrated by a diagram. A study of the color effects produced by dilution shows that the transformation of the  $\beta$ - into the α-form is associated with a depolymerization (Beer's law; see preceding abstr.). This view agrees with the properties of the compds., as given above. The velocity of the transformation of the 2 forms appears to be infinitly great, because "under identical conditions a given salt is never observed to exist in both modifications" simultaneously. [Yet the author gives 2 examples of the simultaneous existence of both forms in the same soln. ABSTR.] J. BISHOP TINGLE.

Formylgiutaconic Ester and its Isomerism Relationships. WILHELM WISLICENUS AND MARGARITA VON WRANGELL. Chem. Lab., Univ. Tübingen. Ann., 381, 367-84.— The preparation of di-Et formyl- (or hydroxymethylene) glutaconate, EtO,CC(: CH-OH)CH: CHCO, Et, has been described previously (Ann., 316, 31 (1901)). At the ordinary temp., when protected from H<sub>2</sub>O, it passes slowly into an oily, dimol. form, the color with FeCl, changing from violet to red. The transformation is hindered by C<sub>a</sub>H<sub>a</sub> and is perhaps prevented entirely if the C<sub>a</sub>H<sub>a</sub> be anhydrous. The presence of H<sub>2</sub>O greatly increases the velocity of the change and a rise in temp. has the same effect. The action of moist Et<sub>2</sub>O is the most convenient method for causing the polym-The formation of the dimol. form, EtO,CCH,CH: C(CO,Et)CH(OH)CH-(CO<sub>2</sub>Et)CH: C(CHO)CO<sub>2</sub>Et, is supposed to be preceded by that of the aldehyde, EtO<sub>2</sub>CC(CHO): CHCH<sub>2</sub>CO<sub>2</sub>Et. The dimol. compd. gives an o.ange-yellow, very hygroscopic potassium salt, but it does not react with Br, HBr, BzCl or PhN<sub>2</sub>H<sub>2</sub>. Copper salt, usually oily; once green crystals were obtained, m. 97°. At 140-50° (12 mm.) the dimol. compd. decomp. into HCO<sub>2</sub>H, AcOEt and tri-Et 1,3,5-benzenetricarboxylate. As the formylglutaconic ester is converted into the dimol. form at 80°, it g ves these same decomp, products at a higher temp. The dimol form and Ph isocyanate produce a compound, C<sub>17</sub>H<sub>22</sub>O<sub>11</sub>N; yellow oil, which could not be distilled. The compds. described below were prepared f.om crystallized (monomol.) di-Et formylglutaconate, unless otherwise stated. Benzoyl derivative, C10H18O3Bz, from BzCl, Et,O and aq. NaOH; colorless needles from Et.O, m. 63.5°. It slowly liquefies. p-Nitrobenzoyl derivative, C10H11O5COC2H, NO2, is prepared in a similar manner to the Bz compd.; white needles from E ,O, m. 117°. Phenyl isocyanate derivative, C, H, O, CONHPh, colorless, rhombic plates from Et<sub>2</sub>O + petroleum ether, m. 74°. These 3 compds. give no color with FeCl<sub>2</sub>. Dibromo derivative, EtO<sub>2</sub>CCHBrCHBrC(CO<sub>2</sub>Et): CHOH, from Br, in CHCl<sub>2</sub>; oily. When distilled at 180° (14 mm.) it produced once a bromo compound, C10H12OaBr; oil; mol. wt., in freezing C2Ha, 297. Its color with FeCl is intensely red. Treatment of the dibromo derivative with an equimol, proportion of

EtOK, in alc. + Et<sub>2</sub>O, converts it into diethyl bromoformylglutaconate, EtO<sub>2</sub>CCBr: CHC(CO<sub>2</sub>Et): CHOH; long, colorless needles from Et<sub>2</sub>O, m. 55-6°. The color with FeCl<sub>2</sub> is deep violet. Copper salt, Cu(C<sub>10</sub>H<sub>12</sub>O<sub>2</sub>Br)<sub>2</sub>. bright, light green, small, microscopic prisms from C<sub>2</sub>H<sub>6</sub>, m. 144-5°. It may be prepared directly from the crude dibromo derivative by prolonged shaking of the CHCl<sub>2</sub> soln. with Cu(OAc)<sub>2</sub> soln. Yield, 80%. Dil. H<sub>2</sub>SO<sub>4</sub> regenerates the bromo ester. Ethyl bromocumalate, formula (I) below, is prepared by distilling di-Et bromoformylglutaconate; colorless prisms

from alc., m. 94-5°. B., aq. KOH hydrolyzes it to furfurane-2,4-dicarboxylic acid. In CHCl<sub>3</sub>, di-Et formylglutaconate adds 1 mol. of Cl, forming an oil, which gives no color with FeCl<sub>3</sub>. Distillation converts it into ethyl dihydrochlorocumalate (II); liquid, b<sub>18 20</sub> 175-80°.

J. BISHOP TINGLE.

Decomposition of Quaternary Ammonium Hydroxides. I. J. v. Braun. Chem. Inst., Univ. Breslau. Ann., 382, 1-49.—In the case of bromocyanogen additive compds. of tertiary amines, it has been shown that the smaller the relative mass of an alkyl group, the greater is its tendency to be eliminated in the form of alkyl bromide. The tendency it eliminate an aryl, or an alkyl containing a double linkage, is even more marked. The relative ease with which the groups are eliminated—in combination with the acid radicle-from quaternary ammonium salts, is in the order allyl, benzyl, Me, Et, Pr, butyl, amyl and Ph. In the case of quaternary ammonium hydroxides, the order of decreasing ease of elimination(?) is allyl, benzyl, Et, Pr, iso-Bu, Bu, Me, isoamyl, Ph. The following numbers represent the % of quaternary ammonium hydroxide, RNMe,OH, which decomp, when heated, so as to give MeOH; only the formula, or name of R is given: Et, o; Pr, 5-10; Bu, 50; amyl, 60; hexyl, 73; heptyl, octyl and cetyl, 75 each. Similar results for the following compds. are given: PhO(CH2)2NMe2OH, 10; PhO(CH2)4NMe2OH, 55; MeO(CH2)2NMe2OH, PhO-(CH,),NMe,OH, H,N(CH,),NMe,OH, BzNH(CH,),NMe,OH, 60 each; PhCH,NMe,OH, slight; Ph(CH<sub>2</sub>),NMe<sub>2</sub>OH, o; Ph(CH<sub>2</sub>), NMe<sub>2</sub>OH, 70; and Ph(CH<sub>2</sub>),NMe<sub>2</sub>OH, 75. The formation of unsaturated compds., when these are produced at all, must be a primary reaction and must be independent of the previous formation of an alc. If this were not so, an Et compd. would give EtOH, which could easily be isolated. The larger proportion of alc. which is produced in the case of the higher alkyl derivs. is due to the difficulty of producing C chains with double linkages at the ends. This difficulty increases as the chain becomes longer. The compds. described below were prepared either by methylating primary amines, RNH2, or by the interaction of Me2N and RBr, or of RI. Butyltrimethylammonium hydroxide, BuNMe,OH, from MeI; viscid liquid. Iodide, purified by means of CHCl<sub>2</sub>, softens 225°, m. 230° (gas evolution). When distilled the hydroxide gives butylene and dimethylbutylamine, BuNMe,; mobil liquid with a basic odor, b. 96°. Yield, 50%. Chloroplatinate, reddish yellow plates, m. 110°. Picrate, reddish yellow plates, m. 98°. Amyltrimethylammonium hydroxide, C,H,1NMe,OH, syrup. When distilled it gives pure propylethylene and dimethylamylamine. Yield, 60%. The propylethylene forms 1,2-dibromopentane, PraCHBrCH2Br; b. 187-90°. In Et<sub>2</sub>O, bromocyanogen and amyldimethylamine form amyltrimethylammonium bromide and amylmethylcyanoamine, CoH11NMeCN; oil with a pleasant odor, b14 109°. Yield, 70% of the parent amine. It is hydrolyzed by b. during 12 hrs., with alc.-H<sub>2</sub>O H<sub>2</sub>SO<sub>4</sub> (30%) to amylmethylamine. Hexyltrimethylammonium hydroxide, CoH12NMe2OH, was prepared from MeI; solid. Iodide, m. 167°. When the hydroxide is distilled it gives pure hexylene (yield, 5% of the iodide) and hexyldimethylamine, CoH13NMe,; liquid

with a penetrating odor, b. 147°. Yield, 73% of the iodide. Picrate, needles from alc., m. 101°. Chloroplatinate, m. 126-7°. Heptyltrimethylammonium iodide, C.H.11-NMe<sub>2</sub>OH, from NMe<sub>2</sub>; crystallin. Iodide, white, unctuous plates from alc. + Et<sub>2</sub>O, softens 143°, m. 145°. Yield, almost quant. The hydroxide, when distilled, gives heptylene, heptyl alc. and heptyldimethylamine, C<sub>7</sub>H<sub>11</sub>NMe<sub>2</sub>; liquid with an intense odor, b. 172°. Yield, 75%. Picrate, opaque, irregularly developed crystals from alc. + Et.O. m. 83°. Chloroplatinate, interlaced, reddish yellow needles from H<sub>2</sub>O, m. 139°. The yield of the amine from Me,NH and heptyl iodide is very poor and that of heptylmethylamime, from heptyl iodide and MeNH, is still worse. Heptylmethylcyanoamine, C, H, NMeCN, was prepared from bromocyanogen and heptyldimethylamine; colorless, rather viscid liquid with a somewhat intense odor which is not disagreeable, b<sub>18</sub> 142°. Yield, 75%. B. with H<sub>2</sub>SO<sub>4</sub> (33%) and alc., during 4-5 hrs., hydrolyzes it to heptylmethylamine, C<sub>7</sub>H<sub>10</sub>NHMe; liquid resembling the above di-Me compd., b. 168°. Picrate, yellow needles from alc. + Et<sub>2</sub>O, m. 97°. Chloroplatinate, orange-colored plates from H<sub>2</sub>O, m. 168°. Carbamide, C<sub>2</sub>H<sub>12</sub>NMeCONH<sub>2</sub>, silvery lustrous plates from Et<sub>2</sub>O + ligroin, m. 100°. Octyltrimethylammonium hydroxide, C.H., NMe,OH, is best prepared from Me,N; solid. When distilled it gives octylene, octyl alc. and octyldimethylamine, C<sub>2</sub>H<sub>17</sub>NMe<sub>2</sub>; b. 194°. Yield, 75%. Picrate, yellow solid from alc., m. 62-5°. Chloroplatinate, long needles from H<sub>2</sub>O, m. 120°. Cetyltrimethylammonium hydroxide, C1. H. NMe. OH, is purified with some difficulty, on account of its tendency to form emulsions with the Ag compds.; solid. Iod.de, interlaced needles from alc., m. 222°. It is prepared from NMe<sub>2</sub>. When heated, the hydroxide decomp. directly, without m., into cetylene, cetyl alc. and cetyldimethylamine, C16H21NMe2, which was purified by means of the picrate; colorless liquid with a characteristic, not unpleasant odor, b<sub>17</sub> 203-5°. Yield, about 75%. Picrate, long needles from alc. + Et<sub>2</sub>O, m. 69°. Ch'orop'atinate, light yellow flocks, m. 83°. γ-Phenoxypropyltrimethylammonium iodide, PhOCH, CH, CH, NMe, I, is best prepared from NMe, lustrous plates from alc., m. 174°. Its hydroxide, when distilled, gives allyl phenyl ether, CH<sub>2</sub>: CHCH<sub>2</sub>OPh, and phenoxypropyldimethylamine, PhOCH, CH, CH, NMe, 8-Phenoxybutyltrimethylammonium iodide, PhOCH, (CH,), CH, NMe, I, was obtained from NMe,; crystals from alc., m. 169°. The hydroxide, when distilled, gives what is probably butylene phenyl ether, CH, CH, CH, CH, OPh, and phenoxybutyldimethylamine, PhOCH, (CH,), CH, NMe,; water-clear liquid with a slightly basic odor, b, 139-40°. Yield, 55%. Picrate, aggregates of needles from alc., m. 108°. Chloroplatinate, oily. S-Phenoxyamyltrimethylammonium iodide, PhOCH<sub>2</sub>(CH<sub>2</sub>), CH<sub>2</sub>NMe<sub>2</sub>I, is prepared from NMe<sub>2</sub>; crystallin, m. 185°. Yield, quant. Hydroxide, oil. When distilled it gives pentenyl phenyl ether, CH<sub>2</sub>: CHCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OPh, and phenoxyamyldimethylamine, PhOCH<sub>2</sub>(CH<sub>2</sub>)<sub>3</sub>-CH<sub>2</sub>NMe<sub>2</sub>; colorless oil with a slightly basic odor, b<sub>11</sub> 149°. Yield, 60%. Picrate, m. 99°. E-Methoxyamyltrimethylammonium iodide, MeOCH, (CH,), CH, NMe, I, from NMe, crystals from alc. + Et,O, m. 123-4°. Yield, very poor. Its hydroxide, when distilled, appears to form  $\alpha$ -methoxy- $\gamma$ -propylethylene, CH<sub>2</sub>: CHCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OMe, and  $\Sigma$ -methoxyamyldimethylamine, the former was not identified and the latter was lost. Hexamethylpentamethylenediammonium iodide, (INMe,CH,CH,),CH, is formed, together with the preceding iodide, and is also obtained from NMe, and diiodopentane; crystals from alc., darkens above 200°, m. 268-73° (gas evolution). Σ-Benzamidoamyltrimethylammonium hydroxide, BzNHCH2(CH2)2CH2NMe2OH, when distilled under reduced pressure, gives α-benzamido-γ-propylethylene, BzNHCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH: CH, (yield, 40%) and Σ-benzamidoamyldimethylamine, BzNHCH, (CH,), CH, NMe, yield of which was stated previously to be 50%, but the author thinks it should be 60%. Σ-Aminoamyltrimethylammonium chloride hydrochloride, HCl.NH<sub>2</sub>CH<sub>2</sub>-CH<sub>2</sub>-NMe<sub>2</sub>Cl, was prepared by hydrolyzing  $\Sigma$ -benzamidoamyltrimethylammonium chloride by means of conc. HCl, at 160°; hygroscopic crystals. Yield, 80%. Chloroplatinate,

red crystals, m. 218°. The hydroxide was prepared by the use of Ag.SO. followed by that of Ba(OH)<sub>2</sub>. When distilled it gives a mixture of H<sub>2</sub>O, NMe<sub>2</sub>, amyleneamine, MeOH and unsym. dimethylpentamethylenediamine; the 2 amylene compds. were separated by treatment with BzCl and alkali, followed by acid. Amylenebenzamide. BzNHCH, CH, CH, CH; liquid, nsol. in acid, b18 200-5°. Unsym. dimethylbenzoylpentamethylenediamine, Me\_NCH2(CH2)aCH2NHBz, liquid sol. in acid, b.a 220-5°. Combined yield, 80%. Amyleneamine, CH<sub>2</sub>: CHCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>, was also separated from the diamine by distillation; colorless, mobil liquid with an intense odor, bree 91-4°. It absorbs H<sub>2</sub>O and CO, from the air. Chloroplatinate, red plates from H<sub>2</sub>O, m. 166° (gas evolution). Chloroaurate, yellow ppt.; when warmed with H2O it deposits Au, softens 180°, m. 195°. Phenylsulfonyl derivative, oily. Methiodide, m. 195°. B-Phenylethyltrimethylammonium bromide, PhCH-CH-NMe-Br, is best prepared from Me,N and phenylethyl bromide. Its hydroxide decomp. even when its aq. soln. is warmed, into NMe, and phenylethylene. 7-Phenylpropyltrimethylammonium hydroxide, PhCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>NMé<sub>2</sub>OH, when distilled, gives  $\gamma$ -phenylpropyldimethylamine, PhCH,CH,CH,NMe, and sym. phenylmethylene, PhCH: CHMe. Yield, 70 and 28%, resp. Σ-Phenylamyltrimethylammonium hydroxide, PhCH<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>NMe<sub>2</sub>OH, from the iodide, gives, when distilled, 2-phenylamyldimethylamine, PhCH<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>-NMe<sub>2</sub>; water-clear liquid with a slight basic odor, b<sub>18</sub> 134-5°. Yield, 75%. Picrate and chloroplatinate, oily. A phenylamylene, PhC<sub>2</sub>H<sub>0</sub>, is formed, together with the preceding amine; water-clear liquid with an intense odor, which is not unpleasant, b.  $197-8^{\circ}$ ;  $d^{20}$  0.8851;  $n_{\rm D}$  1.5064;  $M_{\rm D}$  49.04. Yield, 20%. It decolorizes "perman-I. BISHOP TINGLE. ganate." Dibromo derivative, oily.

Constitution of Morphothebaine. II. Synthesis of the Tetramethoxyphenanthrene which was Obtained by the Degradation of Morphothebaine. R. PSCHORR AND GG. KNÖFFLER. Chem. Inst., Univ. Berlin. Ann., 382, 50-61; see C. A., 4, 2135.— Bensoylamino-2,4-dimethoxycinnamic anhydride, C12H14O4N, is prepared from 2,4dimethoxybenzaldehyde, hippuric acid, AcONa and Ac<sub>2</sub>O; needles from dil. alc., m. 182° (cor.). It dissolves in about 50 pts. of b. alc. After about 5 hrs. b. with aq. NaOH (10%), it is hydrolyzed to NH, BzOH and 2,4-dimethoxyphenylpyruvic acid, it is preferable not to separate this from the BzOH, but to oxidize it directly, in the alkaline soln., by means of H<sub>2</sub>O<sub>2</sub> (3%), at the ordinary temp., during 20 hrs. The resulting mixture of acids is esterified and distilled and the portion b. above 215° is hydrolyzed to 2,4-dimethoxyphenylacetic acid, (HO),C,H,CH,CO,H; needles from Et,O m. 113° (cor.). Yield, 50-60%. Its sodium salt, when heated during 3 ds., at 05-10°, under pressure, with Ac<sub>2</sub>O and 2-nitro-3,4-dimethoxybenzaldehyde, gives \alpha-2',4'dimethoxyphenyl-2-nitro-3,4-dimethoxycinnamic acid, (MeO)<sub>2</sub>C<sub>4</sub>H<sub>2</sub>(NO<sub>2</sub>)CH: C[C<sub>4</sub>H<sub>2</sub>-(OMe), CO,H, which was purified by means of the NH4 salt; yellow tablets, m. 232° (cor.). Yield, 40%. Ammonium salt, plates. It is reduced, at 90°, by aq. FeSO<sub>4</sub> + NH<sub>2</sub> to α-2',4'-dimethoxyphenyl-2-amino-3,4-dimethoxycinnamic acid; yellow cubes from MeOH, m. 207-8°. Yield, about 70%. 3,4,6,8-Tetramethoxyph:nanthreneg-carboxylic acid, formula (I) below, is prepared from the preceding compd. by means

of the diazo reaction; plates from MeOH, m. 226° (cor.). Yield, 30%. It is con-

verted into 3,4,6,8-tetramethoxyphenanthrene (II) by heating with AcOH, at 240°, during 20 hrs. It is identical with the compd. prepared previously from 3,4,6-trimethoxyphenanthrene-8-carboxylic acid (III) by means of the amine. This result not only confirms the formula (III), it also confirms (IV) as being the correct expres-

sion for morphothebaine, because (III) is produced from (IV). 3,4,4'-Trimethoxy-2,2'-oxidostilbene-a'-carboxylic acid (V), is formed, together with (I), from which it is sepd. by its greater solubility in Et<sub>2</sub>O; colorless prisms from MeOH, m. 253°. Methyl ester, m. 182°. 3,4,6-Trimethoxy-8-hydroxyphenanthrene-9-carboxylic lactone (VI), is produced together with (II) and is separated by its insol. in Et<sub>2</sub>O. It crystallizes with difficulty and is best purified by extracting with b. alc.; m. 200-3°. B., alc. NaOH hydrolyzes (VI) to 3,4,6-trimethoxy-8-hydroxyphenanthrene-9-carboxylic acid (VIII); yellow prisms from MeOH, m. 195°. It is converted by Me<sub>2</sub>SO<sub>4</sub> into methyl 3,4,6,8-tetramethoxyphenanthrene-9-carboxylate, see (I) above, which is also formed by methylating (I), or by distilling (VII). In this last reaction the production of the

Me ester is accompanied by that of (VI). When hydrolyzed the ester regenerates (I).

J. BISHOP TINGLE.

Oxidation of 3- and of 7- Methyluric Acid in Presence of Ammonia. OSKAR GROBMANN. Org. Chem. Lab. Techn. Hochsch., Hannover. Ann., 382, 62-81.—Hydroxymethyleneuric acid was prepared from uric acid and HCHO, according to the directions in Ger. Pat. 102,158. Yield, 91%. It is reduced to 7-methyluric acid by means of Sn and HCl. Yield, 50%. 3-Methyluric acid was prepared from uric acid, MeI and aq. KOH (Ger. Pats. 91,811 and 92,310) and was purified in the manner described by E. Fischer and Ach (Ber., 32, 2727 (1899)). Yield, 50%. The oxidizing agent employed in the expts. described below was K<sub>2</sub>Fe(CN)<sub>6</sub>, in presence of aq. NH<sub>8</sub>; it is referred to simply in terms of O. Under varied conditions of temp., 7-methyluric acid is oxidized by 1 or 1.5 ats. of O to a compound, formula (I) or (II) below; lustrous,

hexagonal prisms from H<sub>2</sub>O, decomp. 185-7°. Conc. HCl, or H<sub>2</sub>SO<sub>4</sub>, converts it into a resin and it does not give the murexide reaction. It is sol. in about 240 pts. of cold H<sub>2</sub>O. Yield, 22.3%. It is not converted into an anhydride by recrystallization

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from aq. NH<sub>2</sub>. Prolonged b. with H<sub>2</sub>O hydrolyzes it to NH<sub>2</sub>, carbamide, methylcarbamide, (NH4), C,O4 and a compound, H,MCONMeC(OH)(CO2NH4)NHCONH2; it is sepd., together with a resinous material, by means of its insolubility in a'c.; when the 2 are dissolved in H<sub>2</sub>O and alc. added the resin is converted into (NH<sub>4</sub>)<sub>2</sub>C<sub>2</sub>O<sub>4</sub>, which crystallizes, and the above compound is isolated from the mother liquor. Monoclinic, hexagonal prisms from H<sub>2</sub>O, decomp. 189-2°. In formulating the above NH<sub>4</sub> compd., it has been assumed, quite arbitrarily, to be derived from (I). With aq. KOH, at the ordinary temp., it evolves NH<sub>3</sub>. Silver salt, silky, lustrous plates from H<sub>2</sub>O, darkens 190°, decomp. without m. 204°. At the ordinary temp., HCl hydrolyzes the above compd., C<sub>5</sub>H<sub>12</sub>O<sub>5</sub>N<sub>5</sub>, to carbamide, NH<sub>2</sub> and methyloxaluric acid, the yield of which is 91%. An attempt to recombine these substances to the parent compd., CaH12OaNa, gave only NH, methyloxalurate. When warmed on the H<sub>2</sub>O bath, with HCl (10%), (I) is hydrolyzed to NH<sub>2</sub>, carbamide and methylparabanic acid. When dissolved in 2 N aq. KOH, on the H<sub>2</sub>O bath and immediately cooled, (I) is hydrolyzed to NH<sub>2</sub> and a compound,  $C_bH_{10}O_bN_4$ , which is possibly the acid of the NH<sub>4</sub> salt  $(C_bH_{12}O_bN_b)$ described above; thin needles from H<sub>2</sub>O. It is easily decomp. by H<sub>2</sub>O into the same products as are obtained from the NH<sub>4</sub> salt. The mother liquor obtained in the course of the preparation of (I) (1 at. of O) contains  $K_4$ Fe(CN)<sub>a</sub>,  $\beta$ -methylallantoin. the compd. C<sub>b</sub>H<sub>13</sub>O<sub>b</sub>N<sub>b</sub> and the amorphous substance described above. Similar results were obtained from the mother liquor from the expts. with 1.5 ats. of O, except that no  $\beta$ methylallantoin could be isolated. No definit products could be isolated by the oxidation of 3-methyluric acid in the manner described above, with 1 at. of O, but with 2 ats. of O it gives (I). Yield, 18% of the parent compd. The mother liquor contained the compd. C<sub>b</sub>H<sub>12</sub>O<sub>b</sub>N<sub>5</sub>, described above, carbamide and a sparingly sol. compound, C<sub>4</sub>H<sub>10</sub>O<sub>8</sub>N<sub>4</sub>; bundles of colorless leaflets from H<sub>2</sub>O<sub>1</sub>, decomp. 242-4°. Yield, very small. It gives the reactions of oxaluric acid, but not those of C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>, and with aq. J. BISHOP TINGLE. KOH, at the ordinary temp., it evolves NH,

Behavior of p-Substituted Nitrosobenzenes towards Concentrated Sulfuric Acid. Eug. Bamberger and W. Ham. Analy. Chem. Lab Federal Polytech., Zurich. Ann., 382, 82-128.—Conc.  $H_2SO_4$ , at 20-4°, converts p-chloronitrosobenzene, in glacial AcOH, into a mixture, the main constituent of which is dichlorophenazine N-oxide, formula (I, R=Cl) below; it remains if the acid liquid be poured into  $H_2O$  and the ppt. be extracted with 2 N aq. NaOH. Golden yellow, silky, lustrous needles from glacial AcOH, m. 237.5-8° (decomp.) (bath 225°). In conc.  $H_2SO_4$  the color is bloodred; in conc., yellow HNO<sub>2</sub>, flame-colored; in conc. HCl yellow. It is also formed, at 110-20°, from p-chloroniline, p-chloronitrobenzene and NaOH. Yield, about 4.5% of the chloronitroaniline. The chief product of this synthesis consists of p,p'-dichloroazobenzene. Conc. HCl and Sn reduce (I) to dichlorophenazine (II, R=Cl);

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lemon-yellow, lustrous needles from xylene, m. and darkens 265.5° (bath 245°). Yield, 96.25% of (I). When moistened with conc. HCl it becomes red, its soln. in the acid is yellow. p-Chloro-p'-nitrosodiphenylhydroxylamine, ClC<sub>2</sub>H<sub>4</sub>N(OH)C<sub>2</sub>H<sub>4</sub>NO, is formed together with (I). It is sepd. by its solubility in aq. NaOH and is purified by repeated soln. in aq. NH<sub>3</sub>; greenish bronze-yellow, lustrous plates from acetone, m., darkens and evolves gas 143° (bath 130°). In conc. H<sub>2</sub>SO<sub>4</sub>, aq. alkali, or NH<sub>3</sub>, the color is

red. Aq. NH,Cl and Zn dust reduce the preceding compd. to p-chloro-p'-aminodiphenylamine, ClC\_H\_NHC\_H\_NH\_; flat, colorless, lustrous needles from gasolin, m. 66.5-7°. It volatilizes slowly with steam; in HCl + FeCl, the color is violet, changing quickly to violet-red, the color is discharged if the liquid be b. With AcOH + PbO. the color is violet; with HCl + NaNO, red, changing to yellow. Hydrochloride, lustrous, white crystals. Sulfate, very sparingly sol. a-Naphtholazo derivative, dark red. B. HCl and Zn dust reduce p-chloro-p'-nitrosodiphenylhydroxylamine to p-chloroaniline. A compound, C.H.ONCl(?), is formed together with (I), from which it is sepd. by its solubility in alc. It is purified by adding H<sub>2</sub>O, distilling the ppt. with steam and extracting with ligroin; orange-red, silky lustrous, interlaced needles, softens 158°, m. 159.5-60° (bath 154°). The substance is neutral in character. The following yields of pure substances were obtained by the action of conc. H,SO, on p-chloronitrosobenzene (35 g.) as above; for details see the original paper: (I), (16 g.); (II), (0.25 g.); p-chloro-p'-nitrosodiphenylhydroxylamine, (5 g.); "red compd.," C<sub>2</sub>H<sub>2</sub>ONIC (1.3 g.); p,p'-dichloroazoxybenzene (0.12 g.); HCl (0.4 g.); p-chloronitrobenzene, traces. The crude material weighed 34.5 g. When the H<sub>2</sub>SO<sub>4</sub>, at 30°, is added to the p-chloronitrosobenzene soln. at 40° and the mixture allowed to remain during a wk. the yield of p-chloronitrobenzene is 47% of the nitroso compd. The following compds. were obtained from p-bromonitrosobenzene and conc. H<sub>2</sub>SO<sub>4</sub>, under conditions of preparation similar to those described in the earlier portions of this abstr. Dibromophenazine N-oxide (I, R=Br), golden yellow, silky lustrous needles from xylene. m., darkens and evolves gas 240° (bath 230°). It was synthesized from p-bromoaniline, p-bromonitrobenzene and NaOH, at 110-5°. Its color reactions are similar to those of the Cl compd. Dibromophenazine (II, R-Br), is prepared from the preceding compd., SnCl, and HCl, and is purified by digestion with NHO, (d. 1.41), at the ordinary temp., during 1 hr.; orange-colored crystals from xylene, m. 244.5-5.5° (bath 230°). Golden-yellow crystals are produced by sublimation. In conc. H<sub>2</sub>SO<sub>4</sub> the color is blood-red; in yellow, conc. HNO2, flame-colored; in conc. HCl, red; in SnCl2 + HCl, dark green. A compound, C.H.ONBr, is formed together with the dibromophenazine oxide; cinnabar-red needles from alc., m. 174° (bath 165°). It sublimes in golden yellow needles and is neutral in character. p-Bromo-p'-nitrosodiphenylhydroxylamine, BrC<sub>6</sub>H<sub>4</sub>N(OH)C<sub>6</sub>H<sub>4</sub>NO, yellow powder from acetone + petroleum ether, m. 155° (bath 145°). In alc. + FeCl, the color is greenish brown; in alkalies blood-red. p-Bromo-p'-aminodiphenylamine, BrC<sub>6</sub>H<sub>4</sub>NHC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, is prepared in a similar manner to the Cl compd., which it resembles closely in its reactions; flat, broad, colorless, lustrous needles from petroleum ether, m. 75.5-6°. Hydrockloride, crystallin. Sulfate, m. 229° (bath 220°). p-Bromo-p'-nitrosobromodiphenylhydroxylamine, BrCoH,N(OH)CoH,BrNO, is formed together with the bromonitrosohydroxylamine (see above) and is separated by means of its greater acidic properties; it is pptd. by HCl, from conc., aq. NH, after the monobromo compd.; crystals from acetone + petroleum ether, m., darkens and decomp. violently 130° (bath 125°). In aq. alkalies and NH<sub>2</sub> the color is blood-red. It is reduced by Zn and HCl to the above p-bromob'-amonodiphenylamine. The position of the second Br at. in the hydroxylamine compd. was not determined. The following yields of pure products were obtained from p-bromonitrosobenzene (42 g.) and conc. H<sub>2</sub>SO<sub>4</sub>: dibromophenazine N-oxide (8.2 g.); bromonitrosodiphenylhydroxylamine (5 g.); dibromonitrosodiphenylhydroxylamine (1.4 g.); dibromophenazine (0.3 g.); compd., C<sub>8</sub>H<sub>4</sub>ONBr(?) (1.5 g.). Of the crude material, 39.43 g. were isolated. p-Iodophenylhydroxylamine softens about 70°, then darkens and is not m. 145°. It m. p. was given previously as 104-5° (Ber., 28, 249 (1895)). p,p'-Diiodoazoxybenzene m. 205.5-6° (bath 200°), not 199-9.5° as stated by Gabriel. At 20-5°, but otherwise under the conditions described above p-iodonitrosobenzene and conc. H<sub>2</sub>SO<sub>4</sub> give the compds. described below. Divodo

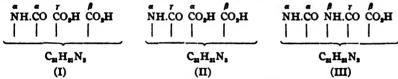
phenazine N-oxide (I, R = I), light brownish yellow, silky lustrous needles from xylene, m., darkens and foams 241° (bath 235°). It is reduced by means of SnCl. + HCl to diiodophenazine (II, R=I); lustrous, yellow needles from xylene, m. 235°, previously softening (bath 228°). In conc H<sub>2</sub>SO<sub>4</sub> the color is bluish green, changing to red on adding a little H<sub>2</sub>O; in yellow, furning HNO<sub>2</sub>, brownish red. The phenazine may be sublimed and, like the oxide, it gives the quinhydrone reaction with SnCl. + fuming HCl. p-lodo-p'-nitrosodiphenylhydroxylamine, IC.H.N OH)C.H.NO, greenish yellow, lustrous plates from acetone. m., darkens and decomp. 150-0.5°. It is only very slightly sol. in aq. NH<sub>2</sub>. Apparently it may be reduced to \$\phi\$-iodo-\$\psi'\-aminodiphenylamine. p-Iodo-p'-nitrosoiododipheny.hydroxylamine, IC,H,N(OH)C,H,INO, brownish yellow, warty aggregates from acetone, m., darkens amd foams 157° (bath 150°). In aq. alkalies and NH, the color is blood-red. When reduced it gives a p-aminodiphenylamine derivative. A compound is also formed together with the diiodophenazine N-oxide; plates with a greenish golden luster and a red color, resembling that of azobenzene, from alc., CHCl2, or ligroin, m. 188.5° (bath 175°). From p-iodonitrosobenzene (45 g.) and conc. H<sub>2</sub>SO<sub>4</sub>, 24.45 g. of crude material were obtained, from which were separated, in a pure condition, p-iodonitrobenzene, (0.07 g.); dijodophenazine N-oxide, (7.1 g.); p-iodo-p'-nitrosodiphenylhydroxylamine, (2.5 g.); p-iodo-p'-nitrosoiododiphenylhydroxylamine, (0.4 g.); red compd., (0.4 g.); diiodophenazine (?) (m. 240°), (0.05 g.). p-Nitrosotoluene, glacial AcOH and conc. H<sub>2</sub>SO<sub>4</sub>, at 20-5°, in the manner described above, give the following compds. Dimethylphenazine N-oxid (I, R=Me), light golden-yellow, silky lustrous needles from ligroin, m. 204-5° (bath 190°). The highly dil., aq., colorless soln. exhibits a pale yellowish green fluorescence, which is discharged by warming, or by adding mineral acids. When heated the base sublimes and s deposited in yellow needles. Hydrochloride, orange-yellow, lustrous needles. When moistened with H<sub>2</sub>O the color changes to S-yellow. Sulfate, resemble the hydrochloride. The oxide is reduced by SnCl<sub>2</sub> + HCl to dime hylphenazine (II, R = Me); very long, light yellow, silky lustrous, or glassy lustrous, highly refractive needles from alc., 162.5-3°. The phenazine may be sublimed; it gives the phenazine reaction and volatilizes slowly with steam the distillate contains about o.o. g. per l. In dil. acids the color is golden yellow. Sulfate, golden yellow needles. The following quantities of the substances were obtained from p-nitrosotoluene (20 g.) and conc. H.SO<sub>4</sub>: dimethylphenazine N-oxide, (4 g.); dimethylphenazine, (0.95 g.); p-azoxytoluene, (0.55 g.); p-azotoluene, (0.05 g.); mixture of the azoxy- and azotoluene, (0.5 g.); amorphous, dark brown powder, (6.5 g.); light reddish brown powder, (2.1 g.); resin. The formation of the monohaloid diphenylhydroxylamines described above necessarily involves the elimination of I halogen at., what happens to it is uncertain. I. BISHOP TINGLE.

Correction. E. Bamberger. Ann., 382, 378.—The "copper salt" formed from Cu(OAc), and nitrosodiphenyhlydroxylamine (Bamberger and Ham, preceding abstr.), is nitrosobenzene.

J. Bishop T ngle.

Chlorophyll. XVI. Primary Transformations of Chlorophyll. RICHARD WILL-STATTER AND MAX UTZINGER. Chem. Lab. Swiss Polytech., Zurich. Ann., 382, 129-94; see C. A., 4, 1472; 5, 2623.—The cryst. chlorophyll used consists of a mixt. of (a) bluish green and (b) yellowish green e hylchlorophyllide, which is produced by the alcoholysis of the total chlorophyll. The material was obtained from Galcopsis, Stachys, Lamium and Heracleum, essential y in the manner described previously. Ethylchlorophyllide (Bo-odin's "crystallized ch orophyll") after having been dried under 0.001-0.01 mm., has the formula C<sub>27</sub>H<sub>39</sub>O<sub>3-3</sub>N<sub>4</sub>Mg. This result indicates that chlorophyllide is composed of chlorophyl.ide (a), C<sub>27</sub>H<sub>39</sub>O<sub>3-3</sub>N<sub>4</sub>Mg and chlorophyllide (b), C<sub>27</sub>H<sub>37</sub>O<sub>9-3</sub>N<sub>4</sub>Mg, or C<sub>27</sub>H<sub>31</sub>O<sub>9-3</sub>N<sub>4</sub>Mg, in the ratio 2.5: 1. Chlorophyllide (a) is regarded

as being made up of approximately equal parts of a lactam, CarHanOaNaMg, and of a lactam hydrate, C<sub>17</sub>H<sub>4</sub> O<sub>2</sub>N<sub>4</sub>Mg, which are probably in combination as a hemihydrate. Dimethyphenylammonium iodide m. 136°, not 124-6° as stated in the literature. The best method for the separation and determination of mixtures of MeI and EtI consists in allowing the jodide to react with Me,N and treating the resulting ammonium jodides with abs. alc. In hot alc. the solubility of Me, NI s 1 g. in 1060 g. and of Me, NEtI 1 g. in 1.23 g. The "AgI number" =  $\omega/S$  100, calculated for ethylchlorophyllide, is 68.0, the values found were 66.1-71.2;  $\omega$  = weight of AgI obtained; S = weight of substance employed. At the ordinary temp., ethylchlorophyllide, in presence of anhydrous MeOH, is converted into a compound, C27H20Q1-1N4Mg.o.5MeOH.o.5H2O; it dissolves in Et<sub>4</sub>O more readily than the parent substance, does not exhibit the brown phase when hydrolyzed with alkalies and is pptd. from Et,O by petroleum ether as a bluish black powder. Alc. reacts like MeOH. In Et<sub>2</sub>O, hydrated (CO<sub>2</sub>H , converts chlorophy lide in 0.2 pheophorbides. The less sol. (A) i deposited in blu sh, or b ownish black, rhombic plates; its color in HCO<sub>2</sub>H is bluish green, in other solvents reddish olive. It does not react with HCl, but is hydrolyzed by alkalies to phytorhodin g and phytochlorin s in the ratio 2.5: 1 (from Galeopsis), 1:1 (from Stackys). The compd. (B) forms long needles, reddish violet-gray by reflected light, light brown in transmitted light. Its solns. are olive-green; in HCO,H blue. Alkali hydrolyzes it to phytochlorin e and phytorhodin g(?), in the ratio 10: 1. The following mol. wt. determinations have been made: Ethylchlorophyllide, CaH4O7N4Mg, in freezing veratrole (K 63.8), 733-42. In b. CHCl<sub>2</sub>: ethylphäophorbide (A), C<sub>17</sub>H<sub>41</sub>O<sub>4</sub>. N<sub>4</sub>, 618-59. Phäophytin, Ca.H., O., N., 802-57. Phylloporphyrin Me ester, Ca.H., O.N., 528-67. Phytochlorin e tri-Me ester, C27HaO2N4. Phytochlorin e Me ester, C2H4O2N, which is also given as  $C_{\text{Bark}}H_{\text{eff}}O_{\text{e}}N_{\text{eff}}$  638-88. Phytorhodin g tri-Me es e ,  $C_{\text{eff}}H_{\text{eff}}O_{\text{e}}N_{\text{eff}}$ , 672-83. Chlorophyll a, in petroleum ether, when treated with KOH, in MeOH (7%), gives a crystallin tripotassium salt, CaHuOaNaMgKa. It liberates chlorophyllin when treated with primary phosphates and gives phytochlorin g with HC. At 140-50°, alc. KOH hydrolyzes the above K salt to glaucophyllin and at 205-10° to rhodophyllin and pyrrophyllin. Chlorophyll, in pyridine, when heated with MeOH and KOH gives an isochlorophyllin, which was isolated as the calcium salt C<sub>24</sub>H<sub>21</sub>O<sub>6</sub>N<sub>4</sub>MgCa<sub>1.6</sub>; light green flocks. The above alkaline KOH soln., by prolonged heating at 225-30°, gives a compd. which is either identical with phyllophyllin, or very closely related to it. Crude chlorophyll, from stinging nettles, when hydrolyzed in hot soln., by aq. Ba(OH), gives phytochlorin e, the above isochlorophyll being an intermediate product. At the ordinary temp., or when heated, conc. alc. KOH hydrolyzes phäophytin and ethylphäophorbide to phytochlorin e and phytorhodin g. This is analogous to the action of hot alkalies on chlorophyll. By the action of conc. alc. KOH on alkylphäophorbides an unstable photochlorin g is formed (acid No. 11) and the feebly basic phytorhodin i, or a similar compd. This corresponds to the hydrolysis of chlorophyll at the ordinary temp. Details are given regarding the isolation of the lactam hydrate and lactam forms of photochlorin e. The hydrate is transformed into the lactam by the action of HCl (4%), at the ordinary temp., during 7 ds. Black, lustrous plates from Et.O, they consist of short, microscopic prisms and plates and are light green, olive-green and brown in transmitted light. Unless completely dried the lactam decomp. slowly into 2 compds., probably photorhodin h and phytochlorin f. The previous statements regarding the solubility of photochlorin s refer to the lactam form. The hydrate dissolves in alc. with difficulty, at the ordinary temp. In conc. H.SO., the color of photochlorin e is bluish green; it is not changed by HCl, but it forms complex barium and potassium salts, which are similar to the na ural Mg compounds. Warm Ac<sub>2</sub>O converts the photochlorin e into a compound with much feebler basic properties (ac d No. 13); bluish black, lustrous, rhombic plates, which are brown in transmitted light. At 140°, quinoline, or diphenylamine, transforms phytochlorin e into phylloporphyrin, but at the temp. of the H<sub>2</sub>O bath photochlorin h is produced; mic ocrystallin aggregates. In Et.O the color is a purer green than is the case with phytochlorin e; in conc. H,SO4, or conc. HCl, green; in dil. HCl, or AcOH, reddish violet; in very dil. NH<sub>1</sub>, olive-green. Acid no. 1.25. The lactam form of phytochlorin e is oxidized by conc. H<sub>2</sub>SO<sub>4</sub> and CrO<sub>5</sub> to methylethylmaleic and hematic imides. New analyses lead to the formulas, C, H, N, CO(CO, H), and C, H, N, C(OH), (CO<sub>2</sub>H), for the lactam and lactam hydrate forms of phytochlorin e. The following additional derivatives of it have been prepared: Tripotassium salt, CuHmO.N.K. or C<sub>2</sub>,H<sub>2</sub>,O<sub>3</sub>N<sub>4</sub>K<sub>2</sub>, by b. the hydrate with alc., MeOH and KOH; voluminous, brown, almost rectangular plates, or spherical aggregates of thin, steel-blue, acute-angled prisms. Cesium salt, CaH22.3OeNeCs2.5, is prepared in a similar manner; lustrous, steel-blue, opaque, acute-angled prisms which are olive-green under the microscope. Ammonium salts, apparently with 1 and 2 NH<sub>2</sub>; unstable. Trimethyl ester, C<sub>27</sub>H<sub>42</sub>O<sub>4</sub>N<sub>4</sub>, from the above K salt and Me<sub>2</sub>SO<sub>4</sub>; long, interlaced, steel-blue prisms from Et<sub>2</sub>O, m. 188-90°, previously softening. With alc. KOH it regenerates phytochlorin c. MeOH, saturated with HCl, converts phytochlorin e into an ester, C<sub>30.5</sub>H<sub>41</sub>O<sub>6</sub>N<sub>4</sub> (2.5 OMe); long, black, lustrous, rhombic plates, softens 200°, m. 204°. Phytochlorin f, C<sub>24</sub>H<sub>24</sub>-O<sub>3</sub>N<sub>4</sub>, has been prepared from the pheophytin obtained from stinging nettles and is separated from photochlorin e and photorhodin g by fractionation with HCl (11%); violet-black, rhombic crystals, which are olive-green by transmitted light. Its composition is not changed by HCl; in this respect it differs from photochlorin e, with the lactam form of which it is isomeric. It is very sparingly sol, except in pyridine and in HCO.H. Acid no. 10. In HCl of this conc. its color is pure blue. It absorbs 2 NH. and retains 0.5 NH<sub>2</sub>. Cesium salt, C<sub>M</sub>H<sub>21.5</sub>O<sub>3</sub>N<sub>4</sub>Cs<sub>2.51</sub> steel-blue, lustrous prisms. Dimethyl ester, C26H31O2N4, or C26H3 O4N4, from the Cs salt and dry Me2SO4; hexagonal, rhombic plates with a blue luster. Acid no. 11.5. Phytochlorin g is rather unstable and is produced by the action of alc. alkali hydroxides on pheophytin, in Et.O; by the action of acids at the ordinary temp., on the salts formed from chlorophyllin and alc. alkalies; by the pheophytin hydrolysis of component a of modified chlorophyll, in petroleum ether-Et,O soln.; in a similar manner from those derivatives of ethylchlorophyllide, which are readily sol. in alc. In Et<sub>2</sub>O the color of phytochlorin g is olive-green; acid no. 10-11. Its most characteristic property is the production of a strongly basic, red compound, when it is evaporated and warmed with alc. This substance gives a blue hydrochloride, which is readily sol. in Et.O. Another decomp. product of phytochlorin g consists of a dark grayish blue substance, which is sol. in Et<sub>2</sub>O and forms a green ammonium salt. At 140-5°, alc. KOH converts phytochlorin g into a dicarboxylic porphyrin, which resembles glauco- and rhodoporphyrin in solubility and in habit, but gives the following characteristic colors: in conc. HCl, greenish blue, with a slight red shade in transmitted light; in dil. HCl, or hot, glacial AcOH, pure blue. Potassium salt, lustrous, rhombic crystals. At 225-30°, alc. KOH converts phytochlorin g into K pyroporphyrin. Phytochlorin f and alc. KOH, at 140-50°, during 7 hrs., give rhodoporphyrin. After more prolonged heating, at 200°, with alkali, the product consists of pyroporphyrin, whereas, under similar conditions, phytochlorin e gives phylloporphyrin. Phylloporphyrin methyl ester, C23H2102N4, is prepared from phylloporphyrin, MeOH and HCl; large, rhombic plates from Et<sub>2</sub>O, long, thin, acute-angled prisms from acetone, they are lustrous reddish violet and reddish brown in transmitted light, m. 224°. Phytorhodin g is formed from component b of the chlorophyll of all the plants investigated, by the alkali hydrolysis of the pheophytin and pheophorbide, when undiluted. It is also produced by the action of acids on the isochlorophyllin of the b series. Photorhodin g, C24H24O7N4, is deposited from Et<sub>2</sub>O in black, lustrous, opaque, hexagonal prisms; it is possible that a hydrated form, CaHas-O<sub>2</sub>N<sub>4</sub>, may also exist and may be produced by the prolonged action of HCl. Tripotassium salt, CaH2107N4K2, from alc. KOH; blackish brown flocks, or aggregates of rhombic plates, which are olive-green in transmitted light. Tricesium salt, CaHai-O,N,Cs, prepared like the preceding compd.; aggregates of rhombic plates. Trimethyl ester, C<sub>2</sub>, H<sub>40</sub>O<sub>2</sub>N<sub>4</sub>, from the dry K salt and MeSO<sub>4</sub>, at the ordinary temp., or, with great difficulty, from MeOH + HCl; rectangular plates and prisms with a black luster from Et<sub>2</sub>O, m. 207-10°, previously softening. In hot, glacial AcOH the color is brownish red; in HCO,H, greenish blue. With alc. KOH it regenerates phytorhodin g. Phytorhodin i, CaHmOaNa, is prepared in a similar manner to the f and g isomers and is separated by its more feebly basic properties; acid no. 15-16; spherical aggregates of brown, or black, microscopic plates from Et.O. Its solns, are brownish red; in HCl, green; in HCO,H, greenish blue. With NH, it behaves like phytochlorin f. Polassium sall, sparingly sol. in H<sub>2</sub>O. At 150°, alkalies convert phytorhodin i into a porphyrin with 2 CO<sub>2</sub>H groups, whereas, at 200°, the product contains only 1 CO<sub>2</sub>H. The former porphyrin resembles rhodoporphyrin, but differs in its colors; reddish violet, lustrous, rhombic plates which are brown in transmitted light; acid no. 3-4. In Et<sub>2</sub>O the color is light red, with a feeble fluorescence; in conc. HCl, blue with a green tinge; in alc. HCl, reddish blue. Hydrochloride, pptd. from conc. HCl by dilution. At 200°, MeOH+ KOH convert photorhodin i into pyroporphyrin. The authors consider that the various isomers described above differ in the position of the NH-CO linkage, or linkages, thus, in the phytochlorin series, the members e, g and f are represented by formulas (I), (II) and (III), resp. Similar formulas are applied to the other series. In



considering the mol. wts. and the corresponding formulas given above, it is to be understood that the compds. represented as having 5.5 ats. of O may be hemihydrates, isomorphous mixtures, or mol. compds. On the other hand, it may be that the formulas should be doubled and 1 H<sub>2</sub>O deducted from 2 mol. complexes, each containing O<sub>6</sub>. Reference is made to the difficulty of detecting the presence of CO<sub>2</sub>H in compds. of high mol. wt. containing N. Thus W. Küster (C. A., 5, 909) concludes that the acidic properties of hemin very probably depend on the presence of 2 CO<sub>2</sub>H groups, whereas O. Piloty considers that they are due to the existence of 2 phenolic OH groups (C. A., 5, 1261). Against this view it is pointed out that the compd. is esterified by means of alc. and HCl and the resulting alkyl derivatives may be hydrolyzed easily. This behavior does not accord with that of phenols. Moreover, Piloty's formula, —CH<sub>6</sub>CH(C)CH(C)OH, is that of a secondary, aliphatic alc. and not of a phenol.

I. BISHOP TINGLE.

Action of Benzoyl Chloride and of Potassium Cyanide on Benzoyloxybenzoic Acids and on Acylated Hydroxybenzoyloxybenzoic Acids. Francis Francis and M. Nierenstein. Chem. Inst., Univ. Bristol. Ann., 382, 194-210.—m-Nitrobenzoyl-m-oxybenzoic acid, O2NC2H4CO2C4H, CO2H, is prepared by warming at 40° a mixture of m-nitrobenzoyl chloride, m-hydroxybenzoic acid and aq. NaOH (10%); short, thick needles from dil. alc., m. 241°. Yield, 81%. m-Nitrobenzoyl-p-oxybenzoic acid is prepared in a similar manner to the preceding compd.; long needles from MeOH, m. 273-4° (decomp. and gas evolution). Yield, 95%. The reaction described below with BzCl, KCN and the various acylated acids, ROR'CO2R"CO2H, mentioned, resulted in the formation of KCl, ROR'COCN and BzOR"CO2H. The acids were separated by means of aq. Na2CO2 and the residual cyanides were hydrolyzed. In this

manner m-nitrobenzoyl-m-oxybenzoic acid and m-nitrobenzoyl-p-oxybenzoic acid gave m-nitrobenzoylformic acid, OaNCaHaCOCOaH, and benzoyl-m- and p-oxybenzoic acids. m-Nitro-p-carbethoxyoxybenzoic acid, EtO2COC4H2(NO2)CO2H, from m-nitro-phydroxybenzoic acid, 1 N aq. NaOH and Et chlorocarbonate, at a low temp., stellate needles from alc. + H2O, m. 117°. Yield, quant. Chloride, from PCla, at the ordinary temp.; long needles from high b. ligroin + CHCl, m. 72°. Yield, 92%. With well cooled, 1 N aq. NaOH and p-hydroxybenzoic acid it gives m-nitro-p-carbethoxyoxybenzoyl-p'-oxybenzoic acid, EtO, COC, H, (NO,) CO, C, H, CO, H; plates from acetone + H.O. short, thick needles from dil. alc., softens 184°, evolves CO., 184-94°, m. 194-6°. Yield, quant. With BzCl + KCN it gives benzoyl-p-oxybenzoic acid and 3-nitro-4hydroxybenzoylformic acid, O2NC2H3(OH)COCO2H; lustrous scales from CHCl2, softens 59°, m. 61°. With FeCl, the color is brown. 3,4-Dicarbethoxyoxybensoic acid, (EtO<sub>2</sub>-CO), C, H, CO, H, is prepared in a similar manner to the Me derivative (C. A., 3, 898); plates from acetone + H<sub>2</sub>O, m. 122-3° (gas evolution). Chloride, crystals from CHCl<sub>2</sub>+ ligroin, m. 55°. With aq. alkali and "o-nitrosalicylic acid" it gives "3,4-dicarbethoxyoxybenzoyl-o'-nitrosalicylic acid" (EtO,CO),C,H,CO,C,H,(NO,)CO,H; lustrous needles from acetone + H<sub>2</sub>O, m. 212-4° (gas evolution). Yield, 62%. The crude acid should be white, otherwise it must be rejected because it contains "o-nitrosalicylic acid," which is extremely difficult to separate. The vague nomenclature adopted by the authors leaves the relative positions of the NO, and OH groups quite uncertain. With BzCl + KCN the preceding acid gives benzoyl-o-nitrosalicylic acid, BzOC<sub>2</sub>H<sub>4</sub>(NO<sub>2</sub>)-CO.H; crystals from alc., m. 117°. 3,4-Dihydroxybenzoylformic acid, (HO),C.H. COCO<sub>2</sub>H, is formed, as the nitrile, together with the preceding compd.; needles from ligroin + CHCl<sub>4</sub> (1 : 2), m. 92°. Its color with FeCl<sub>4</sub> is green. Yield, 41-3%. 3,4-Dicarbethoxyoxybenzoyl cyanide, (EtO,CO), C,H,COCN, was prepared from the chloride described above and KCN; small, lustrous needles from ligroin, m. 38°. Yield, 78%. It is hydrolyzed by conc. HCl to the preceding acid. 4-Carbethoxyoxybenzoyl-p'nitrosalicylic acid, EtO<sub>2</sub>COC<sub>2</sub>H<sub>4</sub>CO<sub>2</sub>C<sub>4</sub>H<sub>4</sub>(NO<sub>2</sub>)CO<sub>2</sub>H, is prepared from "p-nitrosalicylic acid" and p-carbethoxyoxybenzoyl chloride; small prisms from acetone + H<sub>2</sub>O, m. 194-6° (decomp.). With BzCl + KCN it forms p-carbethoxyoxybenzoyl cyanide, EtO.COC.H.COCN; small needles from ligroin, m. 34°. It may be distilled (10-12 mm.). It is hydrolyzed by conc. HCl to p-hydroxybenzoylformic acid, HOC\_H\_COCO\_H; small cubes from CaHa, small needles from CHCla, m. 84°. 5-Benzoyloxy-3,4-diacetoxygallic acid, BzOC,H,(OAc),CO,H, is formed from BzCl, KCN and pentaacetoxygalloylgallic acid (pentaacetoxydigallic acid); small needles from MeOH, m. 178-9°. Galloyljormic acid, (HO), C, H, COCO, H, is produced (as the nitrile) together with the preceding compd.; lustrous needles from dil. alc. (1:2), m. 114-6°. Its color with FeCl, is the same as that given by gallic acid; with KCN, violet; with alkali and O, deep red. The acid reduces Fehling's soln. Tricarbethoxyoxygalloyl cyanide, (EtO<sub>3</sub>-CO), CaH, COCN, from tricarbethoxyoxygalloyl chloride and KCN; small, lustrous needles from ligroin, m. 98°. It is hydrolyzed without difficulty to the preceding acid. 4-Carbethoxyoxybenzoyl-3'-oxybenzoic acid, EtO2COC4H2CO2C4H4CO2H, was prepared from m-hydroxybenzoic acid, aq. KOH and 4-carbethoxyoxybenzoyl chloride; lustrous scales from acetone + H2O, m. 148°. Yield, 98%. 2,4-Dicarbomethoxyoxybenzoyl-4'-oxy-3'-nitrobenzoic acid, (MeO,CO),CaH,CO,CaH,(NO,)CO,H, was obtained in a similar manner to the preceding compd., from 4-hydroxy-3-nitrobenzoic acid; small needles from alc., m. 172° (decomp.). The preceding 2 acids and also benzoyl-poxybenzoic, 4-carbethoxyoxybenzoyl-4'-oxybenzoic and 3,4-dicarbomethoxyoxybenzoyl-4'-oxybenzoic acids fail to react with BzCl and KCN in the manner described above. I. BISHOP TINGLE.

Syntheses by means of Sun Light. I. H. KLINGER. Chem. Lab. Albertus Univ,

Königsberg i/Pr. Ann., 382, 211-21.—The expts. described below were made with glass tubes,  $70 \times 2$  cm., which were drawn out to a capillary and fused. Each tube contained a glass rod with flattened ends, by means of which solid matter could be removed from the glass. The substances under examination filled the tubes to the extent of  $^{2}/_{3}$ . Phenanthrenequinone (10 g.), salicylic aldehyde (6 cc.) and anhydrous  $C_{8}H_{6}$  (50 cc.), after about 20 ds. insolation, beginning in May, gave salicylphenanthrenekydroquinol, formula (I) below; stellate aggregates of white, silky lustrous needles

from C<sub>2</sub>H<sub>4</sub>, or alc., m. 188°. In another expt., with 5 g. of the quinone and a large excess (25 cc.) of the aldehyde, the yield of (I) was quant., after insolation during 5 wks. No trace of (I) was formed by exposure of the mixed constituents to diffused daylight, during 6 wks. Diacetylsalicylphenanthrenehydroquinol (II), from (I) and AcCl; white, silky lustrous, stellate needles from alc., m. 151°, well developed octahedra from C<sub>8</sub>H<sub>6</sub> + alc., m. 143°. The composition of the 2 forms is identical. Dibenzoylsalicylphenanthrenehydroquinol see (II), from (I) and Bz<sub>2</sub>O, at 150-60°; white, flat needles from glacial AcOH and C<sub>2</sub>H<sub>6</sub>, m. 216-7°. In glacial AcOH, well cooled HNO<sub>2</sub> (d. 1.56) converts (I) into phenanthrenequinone and 3,5-dinitrosalicylic acid. Aq. alkalies and NH, also hydrolyze (I) to salicylic acid and phenanthrenehydroquinol, which, in contact with air, soon passes into the quinone. Insolation of phenanthrenequinone (6.3 g.), cinnamic aldehyde (6.8 g.) and anhydrous C<sub>e</sub>H<sub>a</sub> (30 cc.), during 14 dys., gives cinnamylphenanthrenehydroquinol (III); white, silky lustrous crystals from CHCl2, C2H4, or glacial AcOH, m. 193°. It is hydrolyzed to its constituents by air and alkali hydroxides. Acetyl derivative, C12Ha(COAc)CO2CCH: CHPh, from (III) and AcCl; colorless prisms from dil. AcOH, highly lustrous needles from Et<sub>2</sub>O + alc., large, colorless, flat needles from CHCl<sub>2</sub> + petroleum ether (1:4), m. 154°. Dibromo derivative, C<sub>20</sub>H<sub>18</sub>O<sub>4</sub>Br<sub>2</sub>, from Br and the preceding compd., in CHCl<sub>2</sub>; slender, white needles, m. 211°. Benzoyl derivative, C12Ha(COBz)CO2CCH: CHPh, from (III), BzCl and C<sub>2</sub>H<sub>6</sub>, on the H<sub>6</sub>O bath; white crystals from AcOH, CHCl<sub>2</sub>, or C<sub>2</sub>H<sub>6</sub> + alc., m. 218°. Yield, nearly quant. p-Methoxybenzoylphenanthrenehydroquinol (IV), was formed by the insolation, from June to Sept., of phenanthrenequinone (10 g.), p-methoxybenzaldelyde (8 g.) and anhydrous C<sub>6</sub>H<sub>8</sub>, (30 cc.); lustrous, white crystals from C<sub>2</sub>H<sub>4</sub>, CHCl<sub>2</sub>, or best from acetone, violet-white needles from glacial AcOH, m. 232°. Acetyl derivative, from (IV), AcCl and C<sub>2</sub>H<sub>4</sub>, on the H<sub>2</sub>O bath, during 8 hrs.; colorless,

rhombic plates, m. 175°. Benzoyl derivative, from (IV),  $C_0H_6$  and BzCl, at 150°; white needles, m. 193°. Furfurylphenanthrenehydroquinol,  $C_{10}H_{12}O_4$ , is formed in small quantity by the insolation, during 53 dys. (July -Aug.), of a mixture of the quinone (10 g.), furfuraldehyde (6 g.) and anhydrous  $C_0H_6$ ; orange-colored crystals of the holohedral tetragonal system, m. 193°; a : c 1 : 0.3111. The crystals are doubly refractive and rather feebly pleochroic,  $\omega$  reddish yellow,  $\epsilon$  greenish yellow. Accompanying the preceding substance is a second compound; well developed, ruby red, prismatic rods from  $C_0H_6$ , m. 193°. It could not be separated completely from phenanthrenequinone. Insolation of phenanthrenequinone and acetone gave only resinous material, which could not be purified. Chloral and phenanthrenequinone formed a compound in small quantity; light yellow needles, m. 114-6°. It does not contain Cl.

Benzil and BzH gave benzoin, but, in a second expt., a *compound* was produced; crystallin, m. 140°. It is resolved into its constituents by fusion, or by recrystallization from alc. Chloroanil and alc. give tetrachlorohydroquinol when insolated.

J. BISHOP TINGLE.

Photochemical Reactions in Aqueous Solutions. II. ALFRED BENRATH. Chem. Lab. Albertus Univ., Königsberg i/Pr. Ann., 382, 222-35.—In H2O, sulfanilic acid is hydrolyzed to H<sub>2</sub>SO<sub>4</sub> and PhNH<sub>2</sub> by the action of the rays from a Hg lamp. Insolation, from May to Jan., of a mixture of Et<sub>2</sub>O + H<sub>2</sub>O (1:5 vol.) produced no change. Esters are hydrolyzed by light, but the formation of esters under its influence could not be detected. In presence of H<sub>2</sub>O, light causes the hydrolysis of PhBr to HBr and PhOH; of benzoyl chloride to HCl and benzyl alc.; of EtBr to HBr and alc.; of chloroacetic acid to HCl and glycolic acid, this is first oxidized to glyoxylic acid, which is then resolved into CO, and HCHO; of dichloroacetic acid to HCl and glyoxylic acid, which decomp. in the same manner; of trichloroacetic acid to CHCl, and CO<sub>2</sub>, if air be present the CHCl<sub>2</sub> forms COCl<sub>2</sub> + HCl; of CHCl<sub>3</sub> to HCO<sub>2</sub>H, which then decomp. further, the same reaction taking place in dil. or in abs. alc.; of CCl, to CO, and HCl; of chloral hydrate to HCl and glyoxylic acid, which then decomp. into CO. and HCHO. Similar reactions, which have been studied by other chemists, are also mentioned. It is pointed out that alc. does not "protect" CHCl, from decomp., it simply renders the COCl, harmless; the only way of protecting the CHCl, is to keep it in darkness. In H<sub>2</sub>O, FeCl<sub>2</sub> can only withdraw H from compds., thereby causing an indirect oxidation, whereas Fe(NO<sub>2</sub>)<sub>21</sub> in dil. HNO<sub>2</sub>, may cause an addition of O<sub>2</sub> which does not occur if the Fe(NO<sub>2</sub>), be absent. Thus FeCl<sub>2</sub>, H<sub>2</sub>O and MeOH give HCHO, whereas with Fe(NO<sub>3</sub>)<sub>3</sub> and dil. HNO<sub>3</sub>, the products are CO<sub>2</sub> and H<sub>2</sub>O. The results described below show that insolation of the  $\alpha$ - and  $\beta$ -hydroxy acids mentioned, converts them into  $\alpha$ - or  $\beta$ -ketonic acids, which then undergo further change. For expts. of this kind, if made on a fairly large scale, tubes with a capillary opening should be employed so that excessive pressure of the CO, may be avoided.  $\alpha$ -Hydroxypropionic acid gives CO, and AcH, which forms a resin. Pyruvic acid is probably an intermediate product in this reaction; it also gives CO, and AcH. α-Phenyl-α-hydroxypropionic acid decomp. into CO<sub>2</sub> and acetophenone, which was isolated as the phenylhydrazone. Mandelic acid forms CO, and BzH (phenylhydrazone). Citric acid, in presence of FeCl<sub>2</sub>, forms CO<sub>2</sub> and acetone; acetonedicarboxylic acid is probably an intermediate product, but its presence could not be detected. Malic acid gives CO<sub>a</sub> and AcH. Formylacetic acid behaves in a similar manner and is probably an intermediate product in the decomp. of malic acid. Tartaric acid, in presence of FeCl, gives CO., HCHO, glyoxylic acid and glyoxal, which last was isolated as the phenylhydrazine. With the exception of tartaric and citric acids, the author fails to state if FeCl, was employed in the expts. with the above acids. Glyoxylic acid is produced in large proportion by the photochemical oxidation of tartaric acid, by means of Br. Determinations have been made of the change in conductivity caused by replacing the KCl in a soln., by varying equivalent quantities of HCl, the mean error for 1 N solns. was about 3%, the extreme error was about 6% of the mean value. In the case of 0.1 N solns., these errors were 0.6% and 5%, resp. It follows, therefore, that in the case of the reactions which do not involve the production of a volatil electrolyte, the velocity of the change may be followed, with a considerable degree of accuracy, by determining the change in the conductivity, provided that the initial conc. be less than I N. The photochemical reduction of FeCla, by means of alc., was investigated in this manner, the conductivity being determined at the end of definit time intervals. The results confirm those obtained previously and show that the value  $\Delta/t$  rises rapidly and then remains constant;  $\Delta/t = the$  increase in the

conductivity per minute. The photochemical hydrolysis of CHCl<sub>2</sub>, in alc. + H<sub>2</sub>O (I: I), has been investigated in the same manner. The results show that the velocity is not constant, but that it increases with the time and with the change in conc. The simplest explanation of this is the assumption that the HCl acts as a catalyst and that its activity in this respect increases in proportion to its conc. In the absence of a catalyst, AcOMe is hydrolyzed slowly by light, but the results show that the reaction is purely photochemical. The measurements were made by titration. The hydrolysis of chloroacetic acid is also purely photochemical and the HCl which is formed does not increase the velocity of the reaction. The velocity at first increases with the time and then remains constant; the soln. employed was about 0.6 N. As this reaction with chloroacetic acid proceeds rapidly and uniformly, it has been employed in order to det. to what extent the chemically active rays are absorbed by the solvent employed. The app. consisted of 2 concentric quartz cylinders, of which the inner was quite narrow. The solvent under investigation was placed in the space between the cylinders and the soln. of the chloroacetic acid in the inner cylinder. The liquids were stirred continuously and exposed to the rays from a Hg lamp, during 2 hrs. The increase in the cond. of the acid soln, gives a measure of the transparency of the liquid between the cylinders. The following results were obtained in terms of H<sub>2</sub>O: H<sub>2</sub>O, 100; MeOH, 94; alc., 93; glycerol, 56; AcOH, 31; Pr<sup>3</sup>OH, 27; isobutyl alc., 20; isoamyl alc., 14; AcOEt, 8; AcOMe, 5; toluene, 3; CaHe, 3. The high transparency of H<sub>2</sub>O indicates that the quartz apparatus described above may be employed for the determination of the temp. coeff. of reactions, constancy being maintained by allowing H<sub>2</sub>O, of a definit temp., to flow between the cylinders. Preliminary expts. show that the temp. coeffs. are very small. J. BISHOP TINGLE.

Behavior of Mixed Carbonic Esters when Heated. ALFRED EINHORN AND LEO ROTHLAUF. Chem. Lab., Roy. Acad. Sci., Munich. Ann., 382, 237-65; see C. A., 3, 2456.—The phenolcarbonic esters described below, when heated, decomp. in 3 ways: (1) into CO<sub>2</sub> + ROR'; (2) into (RO)<sub>2</sub>CO + (R'O)<sub>2</sub>CO; (3) by interaction with H<sub>2</sub>O into ROH + R'OH + CO<sub>2</sub> (R = alkyl; R' = aryl). The aminocarbonic esters derived from aliphatic and alicyclic alcs. and alkylamines decomp. only according to (2) and (3), under the conditions stated. Methyl guaiacylcarbonate, MeOC<sub>0</sub>H<sub>2</sub>OCO<sub>2</sub>Me, is prepared by adding gradually Me chlorocarbonate to guaiacol, in pyridine, and is purified by treating with aq. HCl, aq. NaOH and H2O, in the order given; oily liquid with an aromatic odor, b<sub>16</sub> 132-4°. Yield, 54.2%. When b. during 24 hrs. it decomp. into CO2, guaiacol, pyrocatechyl dimethyl ether, diguaiacyl carbonate and probably MeOH and di-Me carbonate. Et guaiacylcarbonate, prepared like the Me ester, after b. during 7 dys. gives CO<sub>2</sub>, guaiacyl carbonate, guaiacol and guaiacyl ethyl ether, b. 207-9°, not 213°. Di-Et resorcinyldicarbonate, prepared as above, b. 270-88°; b<sub>10</sub> 174-6° (decomp.). Yield, 108% of the resorcinol, or 54% of the Et chlorocarbonate. B. during 28 hrs. converts it into a resin, together with resorcinyl mono- and diethyl ethers. With 0.5 of the proportion of Et chlorocarbonate, resorcinol and pyridine form ethyl resorcinylcarbonate, HOC, HOCO, Et; plates from gasolin, m. 52-3°, previously softening; b. 274°; b<sub>11</sub> 170-3°. Yield, 70% of the resorcinol. When b. during 7 hrs. it gives resorcinol and resorcinyl ethyl ether. Methyl β-naphthylcarbonate, C<sub>10</sub>H<sub>2</sub>OCO<sub>2</sub>Me, is prepared like the preceding analogs; prismatic needles from gasolin + Et<sub>2</sub>O, m. 42-3°; b. 297-303° (decomp.). Yield, 83% of the Me chlorocarbonate. After b. during 46.5 hrs. it gives  $\beta$ -naphthol,  $\beta$ -naphthyl carbonate and  $\beta$ -naphthyl methyl ether. The yield of guaiacylcarbonyl chloride, MeOC, H, OCOCI, from guaiacol (20 g.), quinoline (20.8 g.),  $C_aH_a$  (40 g.) and  $COCl_2$  in  $C_aH_a$  (20% soln.; 90 g.), at the ordinary temp., is 22 g. On adding to it diethylaminoethanol, in C<sub>0</sub>H<sub>0</sub>, diethylaminoethyl guaiacylcarbonate, MeOC, H4OCO, C, H4NEt, is formed. Yield, 77.6% of the chloro compd. Hydribromide, C14H21O4N.HBr, rhombohedral crystals from acetone, m. 99-100°. Distillation of the preceding ester, in vacuo, converts it into CO, and guaiacylethyldiethylamine, MeOC<sub>2</sub>H<sub>4</sub>OC<sub>2</sub>H<sub>4</sub>NEt<sub>2</sub>; colorless, basic oil, b<sub>10</sub> 148-50°. Yield, quant. Hydrobromide, C<sub>12</sub>H<sub>m</sub>O<sub>2</sub>N.HBr, crystals from acetone, m. 127-8°. The compds. described below were prepared in a similar manner to the preceding guaiacol derivatives, unless otherwise stated. Thymylcarbonyl chloride (yield, 94.6%) gives diethylaminoethylthymylcarbonate, MeC.H.PraOCO.C.H.NEt.; yellow oil. Yield, 80% of the chloride. Citrate, C. H. O. N. prepared in abs. alc.; microcrystallin, m. 90-5°, previously softening. Yield, quant. Hydrobromide, slender needles from alc., m. 160°. Distillation once or twice of the preceding ester, in a vacuum, converts it quant. into thymylethyldiethylamine, MeC.H.PraOC.H.NEt; liquid, b, 126°. Citrate, CaHaO3N, prisms from abs. alc., m. 142-3°. \(\theta\)-Naphthylcarbonyl chloride, C10H7OCOCI, slender, pale yellow needles from gasolin, m. 65-6°; b. 150-2°. Yield, 92% of the \(\beta\)-naphthol. Diethylaminoethyl \(\beta\)-naphthylcarbonate, \(\Cappa\_0\)-H<sub>2</sub>OCO<sub>2</sub>C<sub>2</sub>H<sub>4</sub>NEt<sub>2</sub>, yellow oil. Yield, 112% of the preceding chloride. Hydrochloride, C17HmO2N.HCl, from dry HCl, in Et.O; aggregates of white needles from acetone, m. 141°. \(\textit{\beta}\)-Naphthoxyethyldiethylamine, C10H,OC2H4NEt, from the preceding ester; yellow oil, b18 202°. Hydrochloride, C14H20N.HCl, from ethereal HCl; small plates from alc. + Et<sub>2</sub>O, m. 138-9°. Ethyl chlorocarboxylsalicylate, ClCO<sub>2</sub>C<sub>2</sub>H<sub>2</sub>CO<sub>2</sub>Et, from Et salicylate; colorless oil, b, 144°. Yield, 96% of the salicylate. Ethyl carbethoxydiethylaminosalicylate, Et.NC.H.O.COC.H.CO.Et, yellow oil. Hydrobromide, C18H2O.N.HBr, from HBr, in Et.O; microcrystallin from acetone, m. 106-8°. Ethyl salicylethyldiethylamine, Et,NC,H,OC,H,CO,Et, yellow oil, b, 179-80°. Hydrochloride, needles from AcOEt, m. 112°. Methyl carbethoxydiethylaminosalicylate, Et. NC. H. O. COC. H. CO.Me, almost colorless oil. Yield, 50% of the diethylaminoethaneol. Hydrobromide, prepared from ethereal HBr; warty aggregates of white needles from acetone + Et<sub>2</sub>O, m. 127-30°. Methyl carbethoxydiethylamino-p-oxybenzoate, yellow oil. Yield, 90% of the chloride (described below). Hydrochloride, C<sub>10</sub>H<sub>20</sub>O<sub>4</sub>N.HCl, from ethereal HCl; aggregates of silky lustrous needles from abs. alc., m. 133-4° (decomp.). Methyl diethylaminoethyl-p-oxybenzoate, from the preceding ester (15 mm.); oil, b<sub>18</sub> 186-9°. Hydrockloride, from ethereal HCl; thin, lustrous plates from AcOEt, m. 147°. Methyl chlorocarboxy-p-oxybenzoate, pale yellow needles from ligroin, m. 58°; b<sub>12</sub> 144°. Yield, 84% of the Me p-hydroxybenzoate. Ethyl diethylaminoethyl carbonate, Et. NC. H.O. CO-Et, from diethylaminoethanol and Et chlorocarbonate, in CaHa; colorless oil, ba 91-4°. Yield, 77.6% of the chloro ester. Citrate, C13H27O10N, prepared in alc.; needles from AcOEt, m. 93-8°. When the carbonate is b. during 12.25 hrs. it decomp. into alc., di-Et carbonate, diethylaminoethanol and bisdiethylaminoethyl carbonate dihydrochloride (m. 222°). Menthyl carbonyl chloride, liquid, b, 105-6°. Yield, 91.2%. Diethylaminoethyl menthyl carbonate, Et,NC,H,O,COC,0H,0, colorless liquid, be 179-80°. Yield, 100-33% of the chloride. Hydrochloride, from ethereal HCl; prismatic needles from AcOEt, m. 142°. After b. during 8.5 hrs. it is decomp. into menthol, menthyl carbonate, diethylaminoethanol and its carbonate.

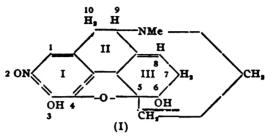
Isocamphane. PETER LIPP. Org. Chem. Lab. Techn. Hochsch., Munich. Ann., 382, 265-305.—On account of the relationship of the sym. compd.,  $C_{10}H_{18}$ , to bornylene, it should be termed bornylene and the asym. isomer camphane. Unfortunately, in the literature the term camphane has generally been applied to the sym. compd., consequently the asym. substance must be distinguished as isocamphane. The camphene employed in the expts. described below consisted of purified com. camphene from pinene hydrochloride;  $b_{784}$  155.3-.5°; m. 45°;  $[\alpha]_{2}^{12}$ —18.94°, in alc., 10% soln. It was not changed by prolonged digestion with Na on a H<sub>3</sub>O bath. At 170-90°, in presence of Ni, it adds 2 H and gives 2 isomers, which were separated by

pressure. The liquid "isocamphene," about 25% of the total, b.  $160-2^{\circ}$ ;  $[a]_{19}^{19}$   $1^{\circ}$ , 15', in MeOH;  $d_4^{90}$  0.8524;  $n_D^{90}$  1.45733;  $R_D$  44.18. The solid isomer is deposited from MeOH in soft, dendritic aggregates, m. 65-7° (cor.). It is optically inactive. Highly purified camphene from isoborneol, when reduced in the manner described above, gave only a solid isocamphane, apparently identical with the preceding compd.; b<sub>700</sub> 164.5° (cor.); m. 61.5-3° (cor.). Yield, of highly purified material, about 5% of the camphene. The liquid "isocamphane," described above, is, therefore, a mixture of the solid isomer with an impurity from the pinene. The same solid isocamphane was formed from camphene and H, in presence of Pt black and Et<sub>2</sub>O; b. 164° (cor.); m. 63-4.5° (cor.). Yield, of pure compd., 24% of the camphane. I-Camphane and Pt black, under similar conditions, gave an optically active isocamphane; crystallin, m. 62-3° (cor.);  $b_{no}$  166-6.5° (cor.); in  $C_0H_0$ ,  $[\alpha]_D^{22}$  8.68°. Both these optical isomers resemble camphene closely in appearance and in odor, but the latter is much less pronounced than that of camphene. The following additional physical properties of the optically inactive isocamphane are given: mol. wt., 141.98 (V. Meyer's method); d47 o.82757; ne 1.43982; np 1.44186; no 1.45239 (cor. for the glass prism), at 67°; Ra 43.99; Rr 45.08; Rp 43.96; Rr-Ra 1.088 The compd. is, therefore, a saturated, bicyclic hydrocarbon. When heated with aq. HI (d. 1.93) (7 pts.), at 240-50°, during 8-9 hrs. and then at 290-5°, during 5 hrs., isocamphane is converted into a mixture of hydrocarbons, but no reduction takes place. Contrary to Sabatier's statement, isocamphane and the liquid isomer are quite stable in air. Isocamphane is attacked only very slowly by CrO<sub>2</sub>, in glacial AcOH, or. in C<sub>2</sub>H<sub>4</sub>, by aq. "permanganate"; in acetone "permanganate" has no action. The best results were obtained by adding finely divided, solid "permanganate" to the hydrocarbon, in warm AcOH (50%). The products consisted of the stereoisomeric camphenilanic acids, camphenilone and camphenilanic aldehyde. Camphenilanalsemicarbazone, C10H10: NNHCONH2, prepared in dil. alc. soln.; large aggregates of colorless plates with 1 H<sub>2</sub>O from dil alc., m. 192° (gas evolution). Yield, 100% of the aldehyde. At 100°, during 8.5 hrs., HNO. (d. 1.4) oxidizes isocamphane to a resin, camphene nitrosite "camphenil nitrite" and camphenilone. The last 3 were removed from the resin by steam distillation, for the details of their separation the original paper should be consulted. When heated on the H<sub>2</sub>O bath, in a reflux apparatus, during 12-3 hrs., with 5 pts. of HNO<sub>2</sub> (d. 1.4), isocamphane gives a mixture of "camphenil nitrite," camphene nitrosite and camphenilone, which are insol. in H<sub>2</sub>O and isocamphoronic and carboxyloapocamphoric acids, which dissolve in H<sub>2</sub>O. The separation and identification of these substances are fully described in the original paper. The formation of carboxyloapocamphoric acid, formula (I) below, involves a rearrangement of the isocamphane (II), the inter-

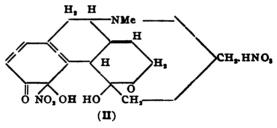
mediate products are probably camphenylic (III) and tricyclenecarboxylic (IV) acids. The above "camphenil nitrite" (Jagelki) is probably a nitrocamphene (V).

J. BISHOP TINGLE.

Morphine. I. HEINRICH WIELAND AND PAUL KAPPELMEIER. Chem. Lab. Acad. Sci., Munich. Ann., 382, 306-39.—E. L. Mayer's "nitrosomorphine" (Ber., 4, 121 (1871)) appears to be morphine nitrite, because it may be prepared by metathesis. 2-Nitrosomorphine, formula (I) below, is prepared by passing the gases from As<sub>4</sub>O<sub>4</sub> +



HNO<sub>2</sub> (d. 1.35) into morphine, suspended in H<sub>2</sub>O, at -2° to -3°, and treatment of the product with aq. NaOH, It is purified by conversion into a salt from which it is regenerated by means of aq. NH<sub>2</sub>, or dil. AcOH; slender, dark orange-red needles with 1 H<sub>2</sub>O from alc., m. 225° (decomp.). The yellow to orange color produced by morphine and HNO<sub>2</sub> may be used as a delicate test for the presence of the alkaloid. The first product of the reaction is a quinol nitrate probably (II), which is unstable.



Sodium 2-nitrosomorphine, C17H18O4N2Na.H2O, is obtained directly from the HNO2 reaction product described above; granit-red needles or plates, from alc. (90%), darkens 220°, not m. 300°. Yield, 60-5%. It is extremely stable towards alkalies. Silver salt, red and pulverulent, black when dry, m. 181-2° (decomp.). No Me deriv. could be prepared from either of these salts. Hydrochloride, C<sub>17</sub>H<sub>18</sub>O<sub>4</sub>N<sub>2</sub>.HCl, from the Na salt; slender, lemon-yellow needles, or large, orange-yellow, rhombohedral granules from dil. HCl, darkens 200°, m. 248° (decomp.). With aq. NaOH the Na salt is regenerated. 2-Aminomorphine, C17H20O2N2, is prepared by the action of Sn and HCl on (I), care being taken to remove N-oxides by a current of air if crude material be employed; lustrous, colorless quadratic plates with H<sub>2</sub>O from alc., m. 258°, previously darkening. It easily passes into a sparingly sol. polymorph, as does also (I) and its Na and HCl salts (see above). In aq. NaOH the color is pale yellow, soon changing to brown. The quantity of Sn required for the reduction shows that (I) is really a nitroso compd. with 1 H<sub>2</sub>O and not a NO, derivative. Hydrochloride, C, H<sub>20</sub>- $O_2H_2.2HCl$ , crystallin. In  $H_2O[\alpha]$ —90°; it has an acid reaction. It reduces Ag-NH, soln. instantly at the ordinary temp. and Fehling's soln. when b.; FeCl, produces a violet color, which quickly changes to olive-green. Yield, about 64% of crude (I). Stannous chloride, cryst. Picrate, slender, yellow needles from H<sub>2</sub>O, decomp. 172°. Diazomorphine anhydride hydrochloride, C17H19O4N.HCl, is prepared at -10°,

from 2-aminomorphine, alc. HCl and EtNO2; yellow, unstable crystals, decomp. about 98°. Treatment with H<sub>2</sub>O and Cu bronze converts it into 2-hydroxymorphine hydrochloride, C17H10O4N.HCl, which could not be purified completely; almost colorless, amorphous powder. It gives a deep brown color with aq. FeCl; its reducing action is similar to that of 2-aminomorphine. Morphine is regenerated by the slow decomp. of the diazoanhydride hydrochloride, in presence of alc. and Cu powder. Benzeneazomorphine, C2H2O2N3, is prepared from morphine, phenyldiazonium chloride and Na<sub>2</sub>CO<sub>3</sub>, it is purified by dissolving in alkali and pptg. with CO<sub>2</sub>; slender, orange-yellow, stellate needles from alc., m. 175° (decomp.). In acids the color is orange-brown, in alkalies dark blood-red. The compd. is reduced to PhNH2 and 2-aminomorphine by means of SnCl<sub>2</sub> and HCl. p-Tolyldiazonium chloride and morphine also give an azo compound, which could not be purified. 2-Aminocodeine, C18H22O2N2, is prepared in a similar manner to 2-aminomorphine, from 2-nitrocodeine; prisms from abs. alc., m. 226°. It gives no color with FeCl<sub>3</sub> and does not reduce Ag-NH<sub>3</sub> solns. Hydrochloride, amorphous. Yield, good. 2-Aminocodeine is readily diazotized and the product instantly ppts. a β-naphtholazo compound: red. When b. with H<sub>2</sub>O, the diazonium salt gives a small yield of 2-hydroxycodeine. C18H2O4N; slender, colorless needles from alc., m. 176°. At 145°, during 1.25 hrs., HCl (30%) converts (I), or its hydrochloride, into 2-nitrosoapomorphine (III); slender, red needles from abs. alc., not m. 300°. In

aq. Na<sub>2</sub>CO<sub>2</sub>, or NH<sub>2</sub>, the color is brownish red: in aq. NaOH, violet with a green tinge. Hydrochloride, C17H16O2N2. HCl, slender, interlaced, yellowish green, silky lustrous needles from H<sub>2</sub>O, darkens about 200°, not m. 300°. It gives an intense olive-brown color with FeCl, and reduces Ag-NH, soln. at the ordinary temp., but does not change Fehling's solution even when b. Aminoapomorphine (see III), is prepared from 2-aminomorphine and HCl (30%), at 130°, during 1.5 hrs.; colorless and amorphous, it quickly becomes violet in air. Hydrochloride, C<sub>17</sub>H<sub>18</sub>O<sub>2</sub>N<sub>2</sub>·2HCl, colorless, voluminous, interlaced needles from dil. aq. HCl, m. 260-5°, previously darkening. Yield, 46.6% of the 2-aminomorphine hydrochloride. The salt gives a deep violet color with FeCla changing quickly to olive-green. It instantly reduces AgNO, soln. at the ordinary temp. and Fehling's soln. when b. morphinequinitrol nitrate (II) is formed, together with (I), but not from it, and is best prepared in a similar manner to (I), without cooling; large, lustrous, orange-yellow prisms. It is decomp. by H<sub>2</sub>O and the base could not be prepared. Yield, 30%. With reagents (II) usually forms amorphous materials. When heated on a H<sub>2</sub>O bath, with H<sub>2</sub>O, during 5-10 mins., it decomp. into NO, CO, HOCN(?) and morphinic acid (IV); crystals from H<sub>2</sub>O. It could not be puri-

fied completely. In H<sub>2</sub>O the color is deep green, changing to cherry-red with HCl. Nitrate, C., H., O.N. HNO, is formed directly from (II); almost colorless, slender needles with 1 H<sub>2</sub>O, from H<sub>2</sub>O, it darkens on exposure to light, or when heated and does not m. Hydrochloride, C17H18O8N.HCl, from the nitrate and conc. HCl; almost colorless needles with 2 H<sub>2</sub>O, from H<sub>2</sub>O. With aq. Na<sub>2</sub>CO<sub>2</sub> the color is carmin-red, changing successively to reddish brown, violet and orange-red. Chloromorphide is best prepared from anhydrous morphine and SOCl, in a freezing mixture; crystals from MeOH, or Et,O, m. 193°. Yield, 90%. Diethylaminomorphide, CuH21O2N2, from the preceding compd. and Et.NH; prisms from abs. alc., m. 203°. Yield, 70% of the Cl compd. Hydrochloride, crystallin, m. 268° (gas evolution). Methiodide, crystallin, m. 268° (decomp.). Bischloroacetylmorphine, CnHalOaNCla, is prepared from anhydrous morphine and chloroacetyl chloride; crystals from Et.O, m. 135°. It is unstable and passes readily into chloroacetylmorphine, C10H20O4NC1; colorless needles from alc., m. 234° (decomp. and evolves gas). Phenylsuljonylmorphinephenylsuljonate, CaHmO.NS.-PhSO<sub>2</sub>H, from morphine and phenylsulfonyl chloride; slender needles from H<sub>2</sub>O, m. 140°. Yield, 125% of the morphine. With Na<sub>2</sub>CO<sub>2</sub> the salt gives phenylsulfonylmorphine, C<sub>12</sub>H<sub>22</sub>O<sub>4</sub>NS; crystals from Et<sub>2</sub>O, m. 165°. It does not dissolve in aq. NaOH, consequently the phenylsulfonyl group is probably linked to the phenolic OH in ring I (see I). In H<sub>2</sub>O, morphinic acid hydrochloride and HONH<sub>2</sub>.HCl form a salt, C<sub>17</sub>H<sub>20</sub>-O<sub>6</sub>N<sub>2</sub>.HCl.H<sub>2</sub>O; crystallin. The preceding compds. have been represented on the basis of Knorr's morphine formula (V), but it is pointed out that morphine may have an

open chain structure (VI). This would accord equally well with all the reactions of morphine and its immediate derivs, and it would make it easier to understand why so many morphine compds. contain "H<sub>2</sub>O of crystn." and why the supposed C-C linkage so readily undergoes scission, in the case of many compds. of this class, such as codeinone, or thebaine.

J. BISHOP TINGLE.

Essential Oil (ROURE-BERTRAND FILS). 17.

Hydrolysis of Metallic Alkyl Sulfates (DRUSHEL, LINHART). 2.

Photochemical Studies (PLOTNIKOV). 2.

Action of Ultraviolet Rays on Saponin (Solacolu). 11.

Solubility Equilibrium between Iodine and Organic Substances (OLIVARI). 2.

Ethereal Oils (Schimmel & Co.) (HAENSEL). 17.

Hydrocarbons of the Butadiene Series (HARRIES). 26.

# 11. BIOLOGICAL CHEMISTRY.

#### WM. J. GIES.

## METHODS AND APPARATUS.

Colorimetric Estimation of Sugar, Creatine, and Creatinine in Urin. W. AUTENRIETH AND G. MÜLLER. Univ. Freiburg. Münch. med. Wockschr., 58, 899-902.—A

modification of the method for sugar previously reported (C. A., 4, 2950). It consists in decolorizing with blood charcoal after addition of Cu soln., thus obtaining complete extraction of the yellow color without loss of sugar. Creatinine is estd. after addition of picric acid and NaOH by comparison with a standard wedge of 0.2 N K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>. Creatine is estd. by conversion into creatinine. G. R. Henry.

New Method of Detecting Salvarsan. P. Beisele. Tutzing. Munch. med. Wockschr., 58, 1313.—Denies specificity of test reported by Abelin (C. A., 5, 3289).

G. R. Henry.

The Direct Determination of Creatine in Pathological Urin. G. S. Wallfolk. Wellcome Physiol. Research Lab. J. Physiol., 42, 801-3.—The pink color given in alk. solns. by creatine but not by creatinine when a trace of diacetyl is added has been made the basis for the quant. estn. of creatine. The presence of NH<sub>8</sub>, arginine, or any protein containing arginine interferes with the reaction. When compared with the method of Folin, sometimes higher, sometimes lower results were obtained. A clinical method is also proposed. For the exact technic see the original article.

I. F. LYMAN.

The Diacetyl Reaction for Proteins. ARTHUR HARDEN AND DOROTHY NORRIS. Lister Inst. J. Physiol., 42, 332-6.—A large no. of complex substances as well as compds. of known comp. were subjected to the test in order to det. the constituent of the protein mol. responsible for the reaction. The test is performed as follows: A dil. soln. of protein or substance to be tested is mixed with a small amt. of 10% KOH soln. and then 1 drop of 1% diacetyl soln. is added. Proteins give a pink color with green fluorescence. Heating in b. H<sub>2</sub>O 1 min. hastens the reaction. Arginine, agmatine, creatine, dicyanamide, and guanidineacetic acid as well as proteins give the pink color but not the flourescence. The reaction depends on the presence of the group NH: C(NH<sub>2</sub>).NH.R. The exact significance of R has not yet been detd.

The Value of Meyer's Reagent for the Examination of Blood. A. SARTORY. Compt. rend., 153, 131-3.—Meyer's reagent (phenolph. reduced by Zn) is not a specific reagent for blood. The typical rose coloration can also be brought about by alkaline bicarbonates. In medico-legal exam. it should only be used to corroborate other tests.

G. M. MEYER.

The Use of the Ester Method in Metabolism Experiments. B. O. PRIBRAM. Z. physiol. Chem., 72, 504.—Additional notes to a previous article (C. A., 5, 2858).

G. M. MEYER.

Test for Hemochromogen (MIRTO). 7.

BACTERIOLOGY.

Bacterial Deterioration of Sugar. 28.

BOTANY.

Formation of Anthocyanin. M. WHELDALE. J. Genetics, 1, 133-57; thru Zentr. Biochem. Biophys., 11, 841.—Mainly theoretical. The action is supposed to go on in 2 sep. stages, each enzymic in nature. Glucoside + H<sub>2</sub>O chromogen + sugar and chromogen + O anthocyanin, the chromogen being colorless. I. K. P.

Presence of Sucrose in the Dried Roots of some Aristolochiaceous Plants and of a Substance Hydrolyzable by Emulsin in the Roots of Asarum europeeum. M. Lesueur. J. pharm. chim., 3, 399-403.—Examined by the Bourquelot biochem. method the dried roots of Aristolochia longa L. were found to contain sucrose. The roots of A serpentaria and of Asarum europeeum contain a small amt. of sucrose and the latter also contain a small quantity of a glucosidal substance hydrolyzable by emulsin. V. K. Chesnut.

Influence of Methyl Alcohol and other Alcohols on Green Plants and Microorganisms. Th. Bokorny. Zentr. Baktr. Parasitenk., II Abt., 30, 53-64.—Different alcohols were added in varying amts. to water and mineral-containing solns. in which green plants, flowering plants, molds, and bacteria were grown. The phenols as a rule do not nourish vegetation, and often have poisonous action. MeOH furnishes C to all forms of plants. EtOH is utilized by bacteria, but not by higher plants. Higher alcohols, except the polyvalent ones, are useless to flowering plants. G. R. Henry.

Resistance of Peroxidases in Heated Grains. JEAN ASPIT AND EDMOND GAIN. Compt. rend. soc. biol., 71, 287-8.—Expts. on wheat grains heated dry and in hot H<sub>2</sub>O showed that the amylase of the grain is destroyed by heat before the peroxidases.

I. K. Phelps

The Mechanism of Carbon Assimilation. III. Francis L. Usher and J. H. Priestly. Proc. Roy. Soc. London, (B) 84, 101-12; cf. C. A., 1, 863.—A correction of views brought forth in the earlier paper and answers to part of the criticisms of A. J. Ewart (Ibid., 80, 30). It is not held now that catalase is localized in the chloroplasts, nor is the post-mortem bleaching of chlorophyll dependent on the presence of CO<sub>2</sub>. The primary products of the photolysis of aqueous CO<sub>2</sub> soln. seem to be CH<sub>2</sub>O and H<sub>2</sub>O<sub>2</sub>. The evolution of O is due to the decomp. of H<sub>2</sub>O<sub>2</sub> by catalase. The processes in the leaf up to this point are non-vital and can be reconstructed in vitro.

I. K. PHELPS.

The Direct Guaiacum Reaction Given by Plant Extracts. M. WHELDALE. Newnham College, Cambridge. Proc. Roy. Soc. London, (B) 84, 121-4.—From expts. with oxidizing enzymes it was found that the power in any plant to give the direct guaiacum reaction is always accompanied by the power to form brown or reddish brown pigment when the tissues are injured mechanically or are subjected to CHCl<sub>3</sub> vapor. The cause of this color change is the oxidation, on exposure to air, of pyrocatechol with the formation of an organic peroxide by which the peroxidase, almost universally present in plants, may transfer O to the guaiacum. The plants examined by the author never give the direct action if pyrocatechol is absent.

I. K. P.

Content of Benzyl Alcohol in Plants. G. CIAMICIAN AND C. RAVENNA. Atti accad. Lincei, 20, I, 392-4; cf. C. A., 4, 1620.—Maize plants were inoculated June 22, July 7 and 19 with a total of 346 g. PhCH<sub>2</sub>OH mixed with an equal amt. of glucose, and harvested July 28, when they weighed 73 kg. The H<sub>2</sub>O ext. contained no PhCH<sub>2</sub>OH, either free or combined, but the AcMe contained 12 g. combined, probably in the form of a glucoside, which, however, could not be isolated in cryst. form.

CHAS A ROUILLIER

Genesis of Alkaloids in Plants. G. CIAMICIAN AND C. RAVENNA. Atti accad. Lincei, 20, I, 614-24.—The amt. of alkaloids in Datura and tobacco plants which had been inoculated with various substances (pyridine, piperidine and carbopyrrolic acid with Datura plants, and, besides these, asparagine, NH<sub>2</sub>, glucose and phthalic acid with tobacco plants) was detd. The inoculated substances disappear almost entirely and the same bases are always obtained, in somewhat greater amt. than in the blanks, pyridine in general producing the greatest increase, altho with tobacco the influence of NH<sub>2</sub> is equally great while that of asparagine is greatest. Simple lesion, without inoculation, also increases the amt. of nicotine; glucose likewise; while phthalic acid seems to cause a decrease. Among the volatil bases from Datura plants were obtained tropine (probably formed from atropine during the manipulations) and possibly tetramethylenediamine, while tobacco yielded isoamylamine. Special expts. showed that this was not formed from protein substances or leucine but is present in the tobacco as a salt or some deriv. easily hydrolyzed even by Mg(OH)<sub>2</sub>.

CHAS. A. ROUILLER.

Resence of Bupleurum fructicosum Linn. L. Francesconi and G. Sanna. Cagliari, ist. chim. gen. r. univ. Gazz. chim. ital., 41, I, 395-414.—Leaves, stems and flowers of Bupleurum fructicosum Linn. from 4 localities (botanical gardens of the Univ. of Cagliari, Monteponi, Buggerru, and Laconi), gathered at different periods (April before blooming, May at the beginning of blooming, June-July in the period of full bloom, July-August after blooming), were distilled with steam. The essence so obtained had a  $d_1$  0.8257-0.8692, decreasing about 0.0008 for 1°,  $[\alpha]$  45.90-19.72°, decreasing with time while the d. increases, n 1.4783-1.4862, b. 167-200°, is oxidized in air and in the light, forming a resinous substance; on long standing or more quickly on heating at 200° protected from the air, it thickens and becomes slightly yellow, the d. increasing and the rotatory power decreasing, and at the same time a viscous substance with negative rotatory power is formed. It increases in amt. up to the period of full bloom, then decreases, the flowers containing the most at the period of blooming. Plants grown at higher altitudes contain more than those grown at lower levels. The d. increases and decreases in the same way as the amt. of essence in the plant; the same is true of the rotatory and refractive powers, except that the former is lower for plants grown at low altitudes. The essence contains no free insol. or difficultly sol. acids. The ester content is greater in the leaves than in the flowers and that of free alcohols less. High altitudes favor esterification, but humidity has an even greater influence in decreasing esterification. The greater the amt. of ester, the greater is the d. and the smaller is the rotatory power. The essence contains practically no substances (phenols) sol. in alk. C. A. ROUILLER.

Chlorophyll (WILLSTÄTTER, UTZINGER). 10.

#### PHYSIOLOGY.

Role of Iron in the Phenomena of Oxidation. Artificial Oxidases with Iron as a Base. J. Sarthou. J. pharm. chim., 3, 49-55.—The author briefly reviews his own and other recent works relative to the similarity between artificial and synthetic oxidizing enzymes, the favoring effect of the Cn-Fe combination in synthetic enzymes, and the subject of co-enzymes and concludes as follows: (1) There is probably no ground for the present distinctions between aeroxidases and anaeroxidases for they are based on insufficient observation, especially with regard to the marked disturbing effect of co-enzymes; (2) an oxidizing enzyme is a catalytic agent formed by the union of a colloid and either Fe or Mn in minute traces; (3) co-enzymes are substances which when present with an oxidase det. a sp. oxidizing action.

V. K. Chesnut.

Contribution to the Chemical Study of the Lipoids of Animal Organs. E. Gerard and M. Verhaeghe. J. pharm. chim., 3, 385-90.—The recently excized organs, freed from adherent fat and other tissue, were dried with sand or plaster and analyzed. The water, P, lipoid, and cholesterol contents, with m. ps. of the latter, are given. To det. cholesterol saponify with alc. KOH, sat. with  $\rm CO_2$ , evap. the EtOH to dryness in presence of sand, and extract with dry  $\rm Et_2O$ . Purify the cholesterol by transforming it into a benzoate by fusion with benzoic anhydride. Dissolve the latter in b. EtOH, cool, collect the purified product and weigh after drying. In all the lipoid masses examined cholesterol oxidation products, especially oxycholesterol, were found.

V. K. Chesnut.

The Examination of Blood. P. JESERICH. Berlin. Ber. pharm. Ges., 21, 222-7; thru Chem. Zentr., 1911, II, 53.—A comprehensive report on the advancement in the field of blood exam. in its chem., physical and physiological relations. E. J. CRANE.

The Cleavage of Glucosides by the Brain. L. HESS. Lab. allgem. Polyklin., Vienna. Wien. klin. Wockschr., 24, 1009–11.—The brains of rabbits, guinea pigs, and man contain a substance which splits  $\beta$ -glucosides, e. g., arbutin, but not salicin

nor  $\alpha$ -methyl-d-glucoside. The brain on being heated lost this cleavage power, indicating the enzymic nature of the reaction. The reaction was favored by a weakly acid reaction, but checked or completely stopped by an alk. reaction. The cerebral fluid also splits  $\beta$ -glucosides, hence the enzyme was sol. in H<sub>2</sub>O. Glycerol exts. were not active.

H. B. Lewis.

The Chemical Processes in the Coagulation of Milk by Rennin. I. Bang. Physiol. Chem. Inst., Univ. Lund. Skand. Arch. Physiol., 25, 105-44; Chem. Zentr., 1911, I, 1709.—The changes produced in milk by the action of rennin for varying periods of time were studied. The Ca saits contained in milk were divided between the organic and inorganic acids, lactalbumin, lactglobulin, and casein. Casein reacted with all bases as an acid. During the coagulation process paracasein gradually joined with increasing affinity to the Ca phosphates, until so much was united with the paracasein that the compd. could no longer remain in soln. Coagulation took place at this point.

H. B. Lewis.

The Viscosity of Body Fluids at Various Temperatures within Physiological Limits.

C. D. SNYDER AND M. H. TODD. Johns Hopkins Univ. Am. J. Physiol., 28, 161-6.

—The viscosity of the blood, plasmata, and sera increases with a fall of temp. The temp. coeffs. of the viscosity of the fluids for intervals of 10 degrees vary with the temp., the larger coeffs. being for the lower, the smaller for the higher ranges.

J. F. LYMAN.

The Meaning of Variation in the Magnitude of Temperature Coefficients of Physiological Processes. C. D. SNYDER. Johns Hopkins Univ. Am. J. Physiol., 28, 167-75.—The temp. coeffs. in many chem. reactions as well as in physiol. actions are variable, tending to grow greater for the lower and smaller for the higher ranges of temp. Sutherland (*Ibid.*, 23, 115) in his theory of nerve conduction has shown that the velocity of the impulse at any temp., within physiol. limits, may be simply a function of the viscosity of H<sub>2</sub>O at that temp. The author does not believe with Sutherland that the viscosity is the only factor that varies the velocity of nerve impulse.

J. F. L.

The Effects of Asphyxia upon Medullary Centers. I. The Vaso-motor Center. G. C. MATHISON. Univ. Coll, London. J. Physiol., 42, 282-300.—The results obtained suggest a common factor, probably the H ion content of the blood, underlying the action of lack of O and of excess of CO<sub>2</sub> upon nerve centers. J. F. L.

 $\beta$ -Imidazolylethylamine, a Depressor Constituent of the Intestinal Mucosa. G BARGER AND H. H. Dale. J. Physiol., 42, 499–503.— $\beta$ -Imidazolylethylamine is present in Popielski's hypothetical "vasodilatin," to which has been attributed the activities of com. peptone and certain organ exts. It has been obtained by the authors from exts. of intestinal mucosa, and is doubtless the substance in Bayliss and Starling's secretin preps. which lowers blood pressure. The base has a very feeble stimulating effect on the pancreas. It does not affect the coagulability of the blood. J. F. L.

Influence of Sex and of Castration upon the Quantity of Lipoids in the Bile of Bovidae.

A. DANIEL-BRUNET AND C. ROLLAND. Compt. rend., 153, 214.—Neither sex nor castration affects the proportion of glycogen and mineral matter in the bile. The lipoids are diminished in the ox.

G. M. MEYER.

Histochemistry of Spermatozoa. H. STEUDEL. Physiol. Inst., Univ. Berlin. Z. physiol. Chem., 72, 305-12.—In all probability, the greater portion if not all of the P in the heads of herring spermatozoa is combined in the nucleic acid. A repetition of the analyses of the spermatozoa gave N values (av. 20.78%) which agree fairly well with those previously obtained by Miescher and by Mathews. P was found to be higher (av. of 6.42 as against av. 5.83, 5.43 and 6.07%).

G. M. MEYER.

Relation between Antithrombin and Autolysis. M. Doyon and A. POLICARD.

Lab. physiol. Fac. méd. Lyon. Compt. rend. soc. biol., 70, 903-4; cf. C. A., 5, 2116.— Experimental data are given which show that antithrombin from the liver of a dog can be isolated by heating the liver immediately after death either on a steam bath or in an autoclave at 110°. Hence, the appearance of antithrombin can not be associated with autolysis as claimed by Conradi.

I. K. Phelps.

Influence of Temperature on the Rapidity of Diastase Reactions. VICTOR HENRI. Lab. physiol., Sarbonne. Compt. rend. soc. biol., 70, 926-7.—The rapidity of the inversion of sucrose by HCl and by invertase is shown to be slightly more influenced by temp. in the case of HCl. This is a further indication that the law of action of diastases is not that of acids.

I. K. Phelps.

Variations in the Production of Secretin in vitro in Macerations of Intestinal Mucous Membrane in Presence of Various Acids. Albert Frouin and S. Lalou. Compt. rend. soc. biol., 71, 189-91.—The quantity of secretin produced by HCl, HI, HNO, H<sub>2</sub>SO<sub>4</sub>, H<sub>2</sub>PO<sub>4</sub>, H<sub>2</sub>C<sub>2</sub>O<sub>4</sub>, trichloroacetic acid, HOAc, citric acid, tartaric acid, and lactic acid was found to be in 0.1 N solns. in proportion to the H ion conc. HI sets free I, consequently its behavior varies from the rule followed by all of the other acids.

I. K. PHELPS.

Influence of Light on the Hyperglobulia of High Altitudes. Tullio Gayda. Atti accad. Lincei, 19, II, 605-11; Chem. Zentr., 1911, I, 672.—The decrease and disappearance of peripheric hyperglobulia in high altitudes when light is excluded is not due to a destruction of the red corpuscles but to an increase in vol. of the plasma which tends to re-establish the normal distribution of the red corpuscles. The author agrees with the views of Foa (Ibid., 12, II, 483) that peripheric hyperglobulia of high altitudes is caused by a stoppage of the blood in the enlarged vessels at the surface, the red corpuscles thus circulating less actively and pptg. in the capillaries. C. A. ROUILLER.

Autolysis of the Brain. FRIEDRICH SIMON. Chem. Abt. Pathologic Inst., Berlin. Z. physiol. Chem., 72, 463-83.—It was found that the protein of calf brain subjected to autolysis undergoes a cleavage due to preëxisting enzymes. About 19% of the organic P is converted to the inorg. sol. form by enzymes. The organic portions of the brain both sol. and insol. in alchohol-ether undergo P cleavage when subjected to autodigestion.

G. M. Meyer.

# PATHOLOGY.

Analysis of Dropsical Fluids. A. LAGENAV. Hospital Militaire, Setif. J. pharm. chim., [7] 3, 489-90.—Analyses of the contents of four alk. tumors containing from 2.5-8.5 l. from 3 different people gave d. = 1.009-1.022; dry residue 20-74, ash 7.4-9.10, serin 4.15-47.5, globulin 2.05-12.25, urea 0.75-2.5, phosphate 0.043-0.3, chlorides 6.43-6.903, sugar 1.4-2.662 g. per l. Only one contained traces of urolilln. Fibrin, fibrinogen, alk, albuminates, mucin, nucleoalbumin, AcOH sol. albumin, albumoses, peptones, indoxyl, biliary pigments, fat, cholesterol, and lecithin were absent.

V. K. CHESNUT.

Analysis of Dropsical Fluid. P. BRETEAU. J. pharm. chim., [7] 3, 248.—Analysis of the alk. fluid from an abdominal tumor containing 20 l. of liquid showed d. = 1.013, dry residue 29.9, ash 8.4, serin 9.75, globulin 9.05, urea 0.8, phosphate 0.05, chlorides 6.7, sugar 0.95 g. per l., with traces of urobilin. V. K. CHESNUT.

The Oxidases of the Blood Corpuscles in Asphyxia. D. MIRTO. Ist. Med. Leg., Univ. Catania. Arch. farm. sper., 12, 119-28.—In asphyxia there is an increase of oxidases in the blood elements, due principally to the immigration of newly formed elements of lymphatic or medullary origin, and perhaps in less degree to the immigration of elements from the vascular walls and to the increased secretory activity of the preëxisting leucocytes.

A. W. Dox.

New Evidence of Causation of Cholera by Nitrous Acid. EMMERICH. Manch. med. Wochschr., 58, 942-9.—Post-mortem exam. of gastric and intestinal contents and of fluid expressed from washed gastro-intestinal mucosa of cholera victims shows acid reaction and HNO<sub>2</sub>. Urin also shows HNO<sub>2</sub>. These findings are pathognomonic. HNO<sub>2</sub> is considered the cause of the severe intoxication as well as of the intestinal and renal lesions. It was not demonstrated in the blood. HNO<sub>2</sub> is formed from nitrates of food by cholera vibrio, and nitrate free diet should protect from intoxication even the the patient harbors organisms.

G. R. Henry.

The Nature of Parathyroid Tetany. A. J. Carlson and Clara Jacobson. Univ. Chicago. Am. J. Physiol., 28, 133-60.—Further expts. show that the increased NH<sub>a</sub> content of the blood in parathyroid tetany (C. A., 4, 2952) does not vary beyond the normal limits. Furthermore intravenous injections of Ca salts sufficient to completely suppress parathyroid tetany do not alter the NH<sub>a</sub> content of the blood. Parathyroid tetany and NH<sub>a</sub> tetany exhibit certain differences in their response to Ca injections and in their behavior after high thoracic transection of the spinal cord. Parathyroid tetany in milder form is suppressed by exts. of hypophysis, hypertonic sugar soln., albumoses, amyl nitrite, section, and stimulation of the vagi (afferent fibers). The authors regard all the measures for the reliet of parathyroid tetany, save transplantation, as temporary palliatives without specific significance.

J. F. LYMAN.

Examination of Urinary Colloids. L. LICHTWITZ. Med. Univ. Klinik, Göttingen. Z. physiol. Chem., 72, 215-25.—In pathologic urin the abs. amt. of colloid is considerably increased and runs parallel with the protein content. Non-protein colloids are present in the urin in the form of sols, gels (reversible) and non-reversible ppts. A ppt. of the colloids is noticed after the use of diuretin and theocine. Protein is present in the urin in a more coarsely divided condition than in blood serum, which condition is not brought about by the urin and is therefore due to the passage of serum thru the kidneys.

G. M. Meyer.

Cerebrospinal Fluid in Pellagra. Pierre Boveri. Compt. rend. soc. biol., 70, 904-6.—A table of results is given of certain measurements of the cerebrospinal fluid in pellagra as follows: color, pressure, surface tension, density, viscosity, reaction of Nonne, reaction of Noguchi, lymphocytosis, and culture exam. I. K. Phelps.

Edema in the Light of Colloid Chemistry. Felix Marchand. Pathol. Inst. d. Univ., Leipzig. Centr. allgem. Path., 22, 625-30.—A discussion of the theory of edema set forth by Martin H. Fischer in his book on that subject. Objection is raised to the explanation of all forms of edema by an increase in the power of tissues to bind water, because in most instances of edema as observed by the pathologist the excess fluid is not fixt in the tissue elements but lies free in the interstices between them; the fibrils of an edematous connective tissue are themselves not at all swollen. Acid intoxication in its most extreme form occurs in diabetes without general edema, and other facts are cited which are not, from the standpoint of the pathologist, reconcilable with this theory.

H. G. W.

# PHARMACOLOGY.

Catalytic Power of Powdered Liver Used in Organotherapy. E. CHOAY. J. pharm. chim., [7] 3, 233-8, 287-92.—The effect of air and heat upon the liver enzymes is like that previously observed by the author in the case of the pancreas (C. A., 4, 2492). An extract dried in vacuo at room temp. showed the same activity towards H<sub>2</sub>O<sub>2</sub> as did the fresh gland; one dried in the air at 42° was only 0.2 as active, while another dried in vacuo at 50° was only 0.8 as strong. The oxidizing action is greatest in the

first half hour, diminishes steadily during the 2nd hr. and very quickly after that and is complete in 24 hrs.

V. K. Chesnut.

The Effect of some Narcotics on Fish. Hans Reuss. Deutscher Fisherei. Neudomm, 1911, 49-57; thru Chem. Zentr., 1911, I, 1606.—Expts. by the author with rainbow trout and common trout show that CHCl<sub>2</sub>, alc., chloral hydrate, paraldehyde, veronal and urethan produce narcosis. All these narcotics, with the exception of urethan, have a more or less strong influence on the respiration centers. Urethan produces with rainbow trout deep narcosis without affecting normal respiration.

E. I. CRANE.

Some Reactions of Surviving Arteries. Douglas Cow. Pharm. Lab., Cambridge. J. Physiol., 42, 125-43; thru Chem. Zentr., 1911, I, 1597.—The effect of different chemicals acting on arterial rings (of man, sheep and ox) suspended in Ringer's soln. was observed by means of a kymograph. CO, by its-action on the muscle tissues of the arterial walls, caused dilation. Alc. (1%) brought about a slight constriction followed by a pronounced dilation. Adreniline soln. (0.001%) has a marked constricting effect on all the arteries except the intravisceral parts of the pulmonary arteries, the coronary and the cerebral arteries. The last two are dilated. BaCla (0.1%) causes a constriction less prompt in appearance, less marked and of shorter duration than that caused by adrenaline. Digitalis constricts the arteries near the surface and those of the organs by acting on the nerves, and paralyses the coronary arteries. Pituitrin (0.1%) causes constriction except in the hepatic, splenic and gastric arteries, which are dilated. Ergotin, tyramine and isoamylamine cause constriction. The ergotin has the most lasting effect. With NaNO, amyl nitrate and sparteine (0.01%) dilation is manifested. Caffeine, urea and Na,SO, cause" Vasodilatation," especially with the visceral arteries. The absence of CaCl, increases the sensitiveness to adrenaline and pituitrin. E. J. CRANE.

The Action of Trivalent Ions on Living Cells and on Colloidal Systems. II. Simple and Complex Cations. G. R. MINES. Cambridge. J. Physiol., 42, 307-31.—The previous work of the author (C. A., 4, 2664 and 2952) which includes a study of salts giving simple ions of the rare earths, La, Vt, Er, Ce, Pr, has been extended to include Nd, Sa, Tm, Gd, Dy, and Yb. These 11 trivalent rare earths have pretty closely the same degree of activity as regards their influence on the frog heart. Sc is much less active, probably because of its lower basicity, its solns, being hydrolyzed, reacting acid and thus resembling Al rather than the rare earths. Five complex trivalent ions [e. g., (Cr(NH<sub>3</sub>)<sub>6</sub>)(NO<sub>3</sub>)<sub>3</sub> yielding the ion (Cr(NH<sub>3</sub>))"] scarcely affected the heart in concs. 100 times greater than the conc. of the simple trivalent ions needed to produce arrest. It is shown by 3 methods that certain negative colloids (emulsoids) have their charge neutralized or reversed with great ease by the simple ions but are unaffected or affected to a much smaller degree by the complex ions. Other colloids (suspended clay) are pptd. as readily by one as by the other kind of ion.

J. F. LYMAN.

Observations on the Role of Potassium Salts in Frog Muscle. W. BURRIDGE. Physiol. Lab., Bristol. J. Physiol., 42, 359–82.—Many compds. produce shortening of isolated frog sartorius and gastrocnemius muscles, e. g., lactic acid, HCl, HNO<sub>2</sub>, H<sub>2</sub>SO<sub>4</sub>, AcOH, alc., CHCl<sub>2</sub>, nicotine, bile, and appropriate concs. of K salts. The action of these substances on muscle is explained on the assumption that they affect the mobilization of the K salts of the muscle. These salts appear to be divided into 2 groups: (1) an indiffusible group situated behind a membrane impermeable to K salts and concerned in the contraction process; (2) a diffusible group situated outside the membrane and more particularly concerned in excitation. J. F. Lyman.

The Effects of Certain Animal Extracts upon the Blood Vessels. J. A. CAMPELL. Dept. Physiol., Edinburgh Univ. Quart. J. Exp. Physiol., 4, 1-17.—Suprarenal ext. in the form of hemisine causes marked constriction of the vessels of all the organs, except the heart and lungs. In the pituitary ext. there are apparently 2 substances, one causing contraction and the other relaxation. Each is capable of producing its characteristic effect on all the arteries except the renal, where contraction is hardly ever obtained, the results being nearly always relaxation.

V. C. Myers.

Action of Poisons on Organic Combustions Studied by their Influence on the Oxidation of Succinic Acid by the Tissues. F. Battelli and L. Stern Compt. rend. soc. biol., 71, 154-6; cf. C. A., 5, 2660.—Such poisons as NaF temporarily check the power of muscle to oxidize succinic acid; such poisons as phenol, acids, alkalies, acetone, formaldehyde, oxalates and persulfate in certain concs. destroy the power of the muscle for oxidizing succinic acid.

I. K. Phelps.

Action of Ultraviolet Rays on the Toxicity of Strophanthin. D. DANIELOPOLU. Compt. rend. soc. biol., 71, 200-1.—From an exposure, varying from 30 min. to 2.5 hrs., of strophanthin to ultraviolet rays its toxicity was very much lessened.

I. K. PHELPS.

Preliminary Note on the Action of Ultraviolet Rays on Saponin. Tr. Solacolu. Compt. rend. soc. biol., 71, 204-5.—Saponin treated for 3.5 hrs. with ultraviolet rays from an elec. lamp loses its hemolytic power. The products after this treatment contained a reducing sugar.

I. K. Phelps.

Action of Alkaloids and their Salts on the Conversion of Starch to Sugar by Amylolytic Enzymes. C. Gerber. Compt. rend. soc. biol., 71, 208-10; cf. C. A., 5. 2660.—The basic salts of quinine accelerate up to a max. conc. the conversion of starch to sugar by diastases (Figuier and Broussonetia). Following this max. these salts exert a retarding influence. The chloride, bromide, valerianate, sulfate, and glycerophosphate of quinine accelerate in all proportions. II. Neutral Salts of Quinine. Ibid., 210-2.—The neutral bromides, chlorides and sulfates of cause acceleration when present in small amts. and in large proportions retard. III. Caffeine, Codeine, their Salts; Salts of Morphine and of Cocaine. Ibid., 212-4.-Caffeine and codeine accelerate in small amts. and have no retarding influence until large amts, are used. Caffeine hydrochloride dissociates to such an extent that its effect becomes like that of HCl. The neutral hydrochloride and monobasic phosphate of codeine accelerate in all proportions. Morphine and cocaine hydrochlorides act like caffeine hydrochloride. I. K. PHELPS.

Action of some Salts on the Conversion to Sugar of Soluble Starch of Fernbach-Wolff by Amylolytic Enzymes. C. Gerber. Compt. rend. soc. biol., 71, 247-9; cf. preceding abstr.—Expts. previously reported, where CdCl<sub>2</sub>, HgCl<sub>2</sub>, K oxalate, Na citrate, ZnCl<sub>2</sub>, CuCl<sub>3</sub>, AgNO<sub>3</sub> and other salts were used, were repeated with the sol. starch of Fernbach-Wolff. The conversion to sugar is as rapid when small amts. of the salts are present as when none is present except for the Hg and Ag salts which act directly on the amylase. In such a case, obviously, conversion to sugar is retarded.

I. K. Phelps.

### ZOOLOGY.

Fluorescence among Luminous Insects. RAPHAEL DUBOIS. Compt. rend., 153, 208-10.—The writer claims that he was the first to discover fluorescent substances in animals, that he established the presence of such substances not only in the Lampyridae and luminous elaters but also in a number of non-luminous animals, and that he suggested the use of fluorescent substances with artificial sources of light to convert useless into useful radiation (cf. C. A., 5, 1607).

F. ALEX MCDERMOTT.

#### MISCELLANEOUS.

Postmortal Pigment Formation. RUDOLF WINTERNITZ. Arch. Derm., 107, 293; thru Zentr. Biochem! Biophys., 11, 846.—On drying skin a dark pigment begins to form after a time; it ceases later. Treatment of the skin with oxidizing or reducing agents in moderate conc. does not hinder the coloring process. An adrenaline-like substance was found several times in rabbit skin.

I. K. Phelps.

Hydrolysis of Protein in Presence and Absence of Glucose. Luciano Pigorini. Inst. Physiol. Ch. Rome. Arch. farmacol., 10, No. 1-2; thru Zentr. Biochem. Biophys., 11, 828.—Cryoscopic detns. were made on the hydrolysis products of protein by alkali and upon the same kind of protein mixed with sugar to det. whether the same products were obtained in presence of sugar as in its absence. The results show that the first cleavage products of protein (proteoses and peptones) combine either with the sugar or with its decomp. products.

I. K. Phelps.

Hydrolysis of Sucrose by Ultraviolet Rays. Henri Bierry, Victor Henri and Albert Ranc. Lab. physiol., Sorbonne. Compt. rend. soc. biol., 70, 900-1.—Solns. of sucrose of different concs. were irradiated for several days at 20° or 40° either under diminished pressure or in contact with the air. Glucose and fructose were first formed, then an osone, CH<sub>2</sub>O, and CO. If CaCO<sub>3</sub> is present, hydrolysis without evolution of gas takes place.

I. K. Phelps.

Influence of Temperature on the Acidity of Proteins and their Derivatives. Ch. Dhirth and S. Sobolewski. Compt. rend. soc. biol., 71, 244-7.—Results are recorded which show in constant vol. the acidity to phenolph. at different temps. of certain monoamino acids, of certain proteins and of certain peptides.

I. K. Phelps.

## 11b. IMMUNOCHEMISTRY.

#### H. GIDEON WELLS.

The Neisser-Wechsberg Complement-Deviation. J. Dunin-Borkowski and M. Gieszczykiewicz. Bull. intern. acad. sci. Cracove, 1910, 746-9; Chem. Zentr., 1911, I, 1145.—Treatment with the same complement quantities and rizing doses of bacteriolytic serums, according to Neisser and Wechsberg (Münch. med. Wochschr., 48, 697), shows a decrease in the lytic strength of the mixture. The expts. of the authors indicate that the cause of the complement-deviation does not lie in the surrounding fluids, where according to Arrhenius (Immunochemie, p. 148) the amboceptor should combine with the complement, but rather solely in the blood corpuscles. They observed that by the use of a considerable conc. of amboceptors the blood corpuscles will undergo a marked agglutination.

Action of Fats on Rabic Virus. CLAUDIO FERMI. Ist. Antirab., Univ. Sassari.

Arch. farm. sper., 11, 260-75.—The fats (olive oil) either by direct action on the virus or by indirect action on the organism completely destroy the immunizing power of antirabic vaccin.

A. W. Dox.

Influence of Substances which Promote or Inhibit Leucocytosis on the Immunizing Power of Normal Nerve Substance. Claudio Fermi. Ist. Antirab. Univ. Sassari. Arch. farm. sper., 11, 194-5.—Aleuronat, which promotes leucocytosis, diminishes the immunizing power of nervous substance more than lactic acid and glucose, which inhibit leucocytosis.

A. W. Dox.

Anaphylaxis. Fritz Munk. Berlin. Med. Klinik, 7, 784-6.—A review.
P. J. Hanzlik.

Toxin of Anaphylaxis. G. Salus and Felix Scellessner. Serolog. Abt. Hygien. Inst. Univ. Prag. Med. Klinik, 7, 970-1.—Authors expts. indicate the presence of a toxin in antisera, but this violent poison cannot with certainty be called an "anaphylatoxin." They also confirm the high toxicity of rabbit-sheep serum.

P. J. HANZLIK.

Criticism of the Side-chain Theory in the Light of Experimental and Literary Investigation. P. Ehrlich and O. H. Sachs. *Münch. med. Wochschr.*, 56, 2529-32, 2586-9.

—A discussion of the work of Bang and Forssman showing that their results are best explained by the side-chain theory.

G. R. Henry.

Is the Ehrlich Side-chain Theory Reconcilable with Facts? P. EHRLICH AND O. H. SACHS. *Münch. med. Wochschr.*, 57, 1287-9.—The authors refute arguments brought by Bang and Forssman against the side-chain theory.

G. R. Henry.

Serum Diagnosis of Trichiniasis. STRÖBEL. Münch. med. Wockschr., 58, 672-4.
—Complement fixation obtained with sera of patients and of infected animals, using as antigen trichinae treated with NaOH and antiformin.

G. R. HENRY.

The Liver in its Relation to Anaphylactic Shock. C. VORGTLIN AND B. M. BERNHEIM. Johns Hopkins Univ. J. Pharmacol., 2, 507-12.—Manwaring's work (C. A.,
5, 529, 1466) was repeated with improved technic, i. e., an Eck fistula was made, the
portal vein clamped near the hilus of the liver and the hepatic artery temporarily
clamped, and hirudin was not used. It was found that no fall of the dog's bloodpressure occurred after the second injection of horse serum when the liver was excluded
from the general circulation. Saline extracts of dog's liver (previously freed from blood)
caused a temporary fall of blood pressure (60 mm.) when injected into normal dogs.
There seemed to be no qual. difference in the action of liver extracts of normal and
sensitized animals. When no anesthetic was used, defecation and urination sometimes followed the injection of the extract, besides muscular weakness and nervous
depression, symptoms found in anaphylactic shock. Boiling destroys this activity
of the liver extract.

The Reliability of the Test for Peptone as a Decomposition Product in Anaphylaxis. F. SCHENE. Pharmakol. Inst., Deutsch. Univ., Prague. Wien. klin. Wockschr., 24, 521-3.—Simple coagulation by heat of normal sera in a faintly acid medium did not yield a filtrate in which the biuret reaction was negative, when the filtrate was first conc. to the original vol. Hence the appearance of the biuret reaction in the serum of anaphylactic guinea pigs cannot serve as a specific indication of anaphylaxis.

H. B. Lewis.

The Effect of Ultraviolet Rays on the Protein Antigen and its Antibodies. R. DOERR AND J. MOLDOVAN. Bakt. Lab. K. K. Militärsanitätskomitees Heilstätte Lepuskranke, Vienna. Wien. klin. Wochschr., 24, 555-9.—Exposure to ultraviolet rays caused a change in the sp. protein bodies of native sera, which depended on the formation of a coagulated irreversible modification, a change which showed itself in the disappearance of sp. precipitability and the loss of the power to produce symptoms in anaphylactic guinea pigs. In protein antisera ultraviolet rays caused a destruction of the pptg. and anaphylactic power. The weakening of the antigenic properties of normal sera and of the immune properties of immune sera resulted uniformly if both the absolute value and the reaction velocity were considered. Exposure of antigens and antibodies for varying periods resulted in the gradual decrease in the reaction velocity, producing the so-called "inhibition zones" (Hemmungszonen). The

latter depend upon a change in the proteins with which the antibodies are associated, rather than upon any change in the immune bodies themselves.

H. B. L.

Researches on the Constitution of Alexin, and Absorption by Specific Precipitates. O. Gengou. Z. Immun. exp. Therap., 9, 344-58.—The middle-piece is bound by specific ppts. After this has occurred the end-piece is also absorbed, but the binding of the end-piece does not take place as readily as that of the middle-piece. Similarly the binding of the middle-piece by sensitized corpuscles takes place more easily than the binding of the end-piece by persensitized corpuscles. Using the proper amts. of corpuscles, it is possible to bind all of the middle-piece of a serum, a part of the end-piece remaining in soln. It would appear that the fresh sera owe their hemolytic and bacteriolytic properties to 2 substances—middle-piece and end-piece—which are not bound together. When there is no middle-piece present, the end-piece is not bound either by specific ppts. nor by erythrocytes.

J. H. MITCHELL.

Vaccination against Pyocyanic Infection by the Intestinal Method. J. Courmount and A. Rochaix. Compt. rend., 153, 131.—Rabbits which had received high colon injections of complete culture of B. pyocyaneus, killed by heat, were protected against 2 cc. of virulent culture.

J. H. MITCHELL.

The Relation of the Dose of Horse Serum to the Protective Dose of Atropine in Anaphylaxis. H. T. Karsner and J. B. Nutt. McManes Lab. Path., Univ. Penn. Proc. Soc. Exp. Biol. Med., 8, 108-9.—The results of Auer and Lewis (cf. C. A., 4, 3097) demonstrating the prophylactic action of atropine sulphate in the asphyxia of immediate anaphylaxis have been entirely confirmed. It has been observed that as the toxic dose of horse serum is increased, the protecting dose of atropine must also be increased, but the increase in protecting dose is not proportionate to that of the horse serum. That the effect of the atropine is physiological and not due to any alkaloidal combination with the toxic fraction of the horse serum is shown by the fact that a mixture of atropine and horse serum incubated at 37° and dialyzed for 4 days killed sensitized animals, whereas a control in the same proportions, but not dialyzed, saved the animals from anaphylactic death.

V. C. Myers.

The Effect of Specific Vaccins in the Typhoid of Rats and Mice. Edna Steinhardt and T. Flournoy. Lab. Hyg. Univ. Mich. and Path. Lab., Bellevue Hosp. Proc. Soc. Exp. Biol. Med., 8, 109-10.—Danysz virus, which produces in rats and mice a disease whose pathology closely resembles typhoid, was employed as the organism. No immunity to the organism could be produced with various vaccins. V. C. M.

Passage of Specific Agglutinin into the Urin in Typhoid Fever. Guido Malan. Inst. Med. Klin., Turin. La clin. med. ital., 49, 290-8; thru Zentr. Biochem. Biophys., 11, 202.—It appears that specific agglutinins do not pass thru into the urin under the usual conditions. Agglutination was never found unless globulin was also present.

I. K. PHELPS.

Contributions to the Study of Anaphylaxis. P. W. Clough. Arb. kais. Gesundh., 31, 431; thru Z. Immunität., Ref., 4, 161.—It is possible to sensitize guinea pigs thru the skin, large intestin, vagina and conjunctiva. It is not necessary for sensitization thru the skin that there be any lesion. Hair dissolved in "antiformin" gave some positive results when used for sensitizing and intoxicating, after neutralization of the solvent.

H. G. W.

Bacterial Anaphylaxis. STUDSINSKY. Inst. Pasteur, Paris. Med. obosrenije, 75, 376; thru Z. Immunitāt., 4, 162.—Both passive and active bacterial anaphylaxis are not as constant or specific as serum anaphylaxis.

H. G. W.

The Biological Place of Lens Proteins and of the Ectodermal Appendages. F. F. KRUSIUS. Univ. Augenklinik, Marburg. Arch. Augenheilk., 47, 47; thru Z. Immuni-

tot., 4, 1247.—An investigation of the source of the organ specificity of lens proteins and the other proteins of ectodermal origin. The lens of squids and pigs contain anaphylactogens which are not biologically very closely related, as they contain only traces of proteins which inter-react. Likewise a certain degree of interaction can be obtained with the proteins of ox lens and the proteins of the lenses of squids or cod fish, but quant, studies show that of the protein of the cod lens less than one-tenth, and of the squid lens protein less than 1/20, is homologous with the ox lens protein; but at least 1/4 of the protein of the lens of these 2 marine animals is identical. The organ specificity of the lens is found to be limited to the central part of the lens, and is only of relative specificity, the cortical portion of the lens shows species specificity like other organs and tissues. Horse hoof proteins make guinea pigs anaphylactic, and contain a protein biologically identical with horse serum. Long-continued action of "antiformin" on protein destroys its antigenic property; eventually there result denaturized proteins which interact with one another irrespective of the biologic origin of the proteins. There is some interaction between the proteins of lens and of the keratinous structures. H. G. W.

Toxopeptides. III. Fr. KEYSSER AND M. WASSERMANN. Kgl. Inst. Infektionskr., Berlin. Fol a serol., 7, 593-605.—The name "toxopeptide" is given to the poison which causes anaphylactic intoxication, and which is formed in the mixt. of antigen-amboceptor-complement by the action of the complement upon protein-like substances which have been adsorbed by the antigen in a purely physical manner. This view is based upon the fact that for the usual organic antigens there may be substituted in such mixts. an inorganic substance, such as BaSO, or kaolin, and yet the toxopeptide will be formed by the amboceptor and complement. Without the adsorptive substance, whether antigen or inorganic adsorbent, no toxopeptide is formed. The toxopeptide is believed to be formed from the amboceptor which has thus been physically adsorbed. The same process can be demonstrated both in vivo and in vitro. Thus, if kaolin and inactive horse serum are injected into guinea pigs or white mice, after a few hours of digestion to permit of adsorption, the normal complement will split off the toxopeptide from the adsorbed amboceptor of the horse serum, and death takes place with the symptoms of anaphylaxis. Also if kaolin acts in the incubator upon the serum of guinea pigs which have been immunized to horse serum, and therefore rich in amboceptors, toxopeptide will be formed by the action of the normal complement upon the absorbed immune amboceptors. As in this last case the combination involves guinea pig amboceptors and guinea pig complement, the possibility of formation of poison from protein by the complement is excluded, supporting the claim that it is the amboceptor itself from which the toxopeptide is formed by the complement. H. G. W.

The Specific Substances of B. tuberculosis and other Acid-fast Bacilli. Orro Deilmann. Inst. Exp. Therap., Eppendorfer Krankenhaus. Z. Immunitat., 10, 421-39.—A corroboration and amplification of the work of Much, who claims that tubercle bacilli and the other acid-fast bacilli have common antigens differing quantitatively, which fact is interpreted as supporting the hypothesis that B. tuberculosis is derived from acid-fast saphrophytes. According to the results of quant. expts. with the complement fixation reaction the leprosy and acid-fast bacilli of urinf are more closely related to B. tuberculosis than the saphrophytic timothy hay bacillus and the blind-worm tubercle bacilli. Several of the constituents of B. tuberculosis give specific reactions, namely the proteins, the free fatty acids, and the neutral fats (tuberculonastin). With tuberculosis serum the strongest specific complement fixation reaction is given by the tuberculo-nastin, next the fatty acids, and much less by the bacil-

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lary protein. The sum of their reactions is about equal to the total reaction of tuberculin or entire bacterial substance.

H. G. W.

Anaphylaxis. XX. The Significance of Quantitative Relations in Anaphylaxis Experiments with Particular Relation to Bacterial Anaphylaxis. E. FRIEDBERGER AND S. MITA. Pharmakol. Inst., Univ. Berlin. Z. Immunitāt., 10, 453-78.—It was found that the results were no less marked in bacterial anaphylaxis than in serum anaphylaxis if the quantities of bacterial protein used for both injections were only somewhat corresponding to those used for serum injections. It was found possible to secure passive anaphylaxis with goat serum (contrary to Kraus). H. G. W.

The Nature of Complement. L. v. LIEBERMANN AND B. v. FENYVESSY. Hyg. Inst., Univ. Budapest. Z. Immunität., 10, 479-502.—It was found possible to obtain mixts. of Na soaps with certain constituents of normal serum, which behaved like natural complement to the extent that they hemolyzed sensitized red corpuscles much more strongly than normal corpuscles; indeed under certain conditions the sensitized corpuscles might be entirely hemolyzed while the normal corpuscles were not hemolyzed at all. The most effective combination was found to be one containing, in addition to the soaps, serum globulin and CaCl, in a medium of a certain definit degree of alkalinity. Such artificial complement may be separated into two fractions corresponding to the middle- and end-piece of natural complement. The complement-fixation reaction could not be duplicated with this artificial complement. H. G. W.

The Specificity of Phytagglutinins. H. RAUBITSCHEK. Czernowitz. Z. Immunitát., 10, 538-40.—Controversial. H. G. W.

# 12. FOODS.

### W. D. BIGELOW.

Record of the Association of Agricultural Experiment Stations. Anon. Z. offent. Chem., 17, 192-5.—Mainly polemical. A protest against the methods used by the control labs. in the exam. of cattle feed.

C. P. WILSON.

Report of the Official Laboratory of the Neusz District for 1910. Kraus. Chem. Zig., 35, 855.—A report of the exam. of meat, fish, butter, honey, fruit syrup and beer.

C. P. Wilson.

Order of the Aus'rian Minister of the Interior Concerning the Austrian Alimentary Codex. Anon. Veröffent. Kais. Gesundh., 35, 718.—Announcement of the first vol. of this book giving rules and regulations on the sampling, investigation, judgment, sale and valuation of foods.

R. F. Bacon.

Order of he Hungarian Minister of the Interior on the Trade in Artificial Sweeteners, Saccharin, etc. Anon. Veröffent. Kais. Gesundh., 35, 742.—Artificial sweeteners such as saccharin, etc., are prohibited in food products except in certain instances allowed on the opinion of the official physician. Regulations for the sale of these substance are given.

R. F. BACON.

Determination of Malic Acid. P. B. DUNBAR AND R. F. BACON. U. S. Dept. Agr., Bur. of Chem., Circ. 76.—The principle used in this method is the fact that the optical rotation of malic acid is greatly increased by the addition of U salts. It is not necessary to sep. the malic acid for this detn., as d-tartaric acid is only the common substance which interferes. The method is as follows: Place 75 cc. soln. in a 100 cc. measuring flask, neutralize, and if dark colored add 5-10 cc. Al(OH)<sub>2</sub> cream, then make up to 100 cc. Filter, treat 25 cc. of the filtrate with powdered UO<sub>2</sub>(C<sub>2</sub>H<sub>3</sub>-O<sub>2</sub>)<sub>20</sub> adding enough of this salt so that a small amt. remains undissolved. Let stand

2 hrs. with frequent shaking, filter and polarize in a 200 mm. tube, thus obtaining reading (1). Clarify the remainder of the filtrate with powdered Pb(C<sub>2</sub>H<sub>2</sub>O<sub>2</sub>)<sub>2</sub>. Filter and remove the excess of this salt with anhydrous Na<sub>2</sub>SO<sub>4</sub>. Filter and polarize (reading 2). The algebraic difference between readings (1) and (2) times 0.036 equals the malic acid in g. per 100 cc. of the polarized soln. If reading 2 is negative and the soln. contains more than 10% sugars and less than 0.25% malic acid treat a portion of the filtrate from soln. pptd. by Pb(C<sub>2</sub>H<sub>2</sub>O<sub>2</sub>)<sub>2</sub> with UO<sub>2</sub>(C<sub>2</sub>H<sub>2</sub>O<sub>2</sub>)<sub>2</sub> as given above and polarize. This reading (3) is used instead of (2), because the reaction of U salts on the polarization of invert sugar solns. is sufficient to vitiate the results where a very large amt. of sugar and very small amt. of malic acid are present. The most favorable limits of conc. of malic acid are 0.2-2.5%. The % error seldom amts. to more than 5% of the malic acid present; 12 detns. may be made in 4 hrs. R. F. BACON.

A Polariscopic Method for the Determination of Malic Acid and its Application in Cane and Maple Products. P. A. YODER. J. Ind. Eng. Chem., 3, 562-74.—Using 1% malic acid solns. the author found for "the U salt malic acid complex"  $[\alpha]^{20}$  = 515,  $[\alpha]_{\rm D}^{20} = -501$ . The conc. of the malic acid up to 1.3% seemed to have no influence. Over 1.25 atoms U to 1 mol. malic acid should be used to get the max. rotation. In pure solns, malic acid may be estd, by the increase in polarization due to the presence of U salts. 3% sugar had no effect on the rotation of "U salt malic acid complex." If neutral, active or inactive substances and other acids are present only in small quantities, proceed as follows: Acidity properly to obtain max. rotation, as follows: Titrate with o. 1 N KOH to a certain shade of methyl orange (see original) and add 0.046 g. cryst. UO<sub>2</sub>(C<sub>2</sub>H<sub>2</sub>O<sub>3</sub>)<sub>2.2</sub>H<sub>2</sub>O for each cc. 0.1 N KOH required for neutralization to phenolph. Polarize in 200 mm. tube at 20° and also the same soln. without the U salt. Multiply the difference in the 2 readings (degrees Ventzke) by 0.0338 if white light was used and by 0.0347 for yellow light to obtain the % of malic acid. In case a large amt. of sugars, acetic or nitric acids are present the malic acid is first sepd. as Pb salt, and the malic acid obtained from this by H<sub>2</sub>S before proceeding as above. 1.5 vol. 95% alc. added is not sufficient to ppt. all the Pb malate in the presence of sugars; 3.6 vols. gave a satisfactory pptn. Using Ba acetate a practically complete pptn. was obtained by the addition of 14 vols. 95% EtOH. In cane and maple sugars and syrups malic acid is sepd. by the Pb or Ba pptn. and then detd. as above. The same method could be applied to the polariscopic detn. of tartaric acid.

R. F. BACON.

Effect of Nitrates and Nitrites on the Turmeric Test for Boric Acid. T. M. PRICE AND E. H. INGERSOLL. U. S. Dept. Agr., Bur. of Chem., Bull. 137, 115-6.—Nitrate was found not to influence the reaction and the effect commonly attributed to nitrates was found to be due to the presence of nitrites. A paler tint on the turmeric paper was given by boric acid in the presence of 0.01% KNO<sub>3</sub>, while 0.1% completely inhibited it. The difficulty may be remedied by adding urea to the soln. when as much as 0.3% KNO<sub>3</sub> does not interfere with the color reaction, even though as little as 0.01% of boric acid may be present. The following method is employed: Acidify 9 cc. of the soln. to be tested with HCl, add 1 cc. of conc. HCl containing 5 g. of urea per 100 cc., shake the mixt., and allow to stand for 5 min. Immerse turmeric paper in this soln. momentarily, hang up, and allow to dry in the air.

W. D. Bigriow.

Exact Method for the Determination of Citric Acid in Citrates and Lemon Juice.

MATTEO SPICA. Catania, ist. agrar. sic. Valdisavoia. Gazz. chim. ital., 41, I, 454-69; cf. C. A., 5, 541.

CHAS. A. ROUILLER.

The Differentiation of Natural and Artificial Fruit Essences. I. A. LANDOLT. Chem. Zig., 35, 677-8.—Exam. was made of a number of artificial fruit essences prepared in the lab., and of a number of natural fruit flavors prepared by macerating

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fresh fruit with alc. or sugar. The detns. made were: sp. gr. of the essence. sp. gr. of the 80% distillate, ester content of distillate, and volatil acid obtained by acidifying the residue from the ester detn., distilling in a current of steam and titrating. Raspberry, strawberry and lemon flavors were used and in each case the analysis readily disclosed the identity of the sample. II. Ibid., 687-8.—Some investigations were made on the method of Declaux for the detn. of AcOH, propionic, butyric and valeric acids alone and in mixts. The method consists in distilling 110 cc. of a 1-2% soln, of the acid, collecting each 10 cc. separately and titrating. For a given acid the acidity of any given fraction is practically constant and hence the total amt. of acid distilled over at any one point in the distillation is practically constant. For a mixt, of 2 acids the acidity of any fraction or the total acidity at any point in the distillation is the arithmetical mean between the corresponding figures for the 2 acids. Some curves are given in which the fractions are represented by abscissas and the \% of total acid distilled by ordinates. A characteristic curve is obtained for each acid. Artificial essences give curves distinctly different from those derived from natural fruit essences. C. P. WILSON.

The Bleaching of Olive Oil. Communication from the French Consul at Florence to the Minister of Foreign Affairs. Ann. jals., 4, 355.—It is held that the natural color of olive oil is golden-yellow, whereas the trade demands an unusually light color. To secure this, bleaching is resorted to. This is best accomplished by thoroly mixing 100 kg. of oil with 3 kg. of tannic acid, dissolving in 25 l. of water and allowing to stand until the 2 liquids separate.

W. D. BIGELOW.

Report on Colors. W. E. MATHEWSON. U. S. Dept. Agr., Bur. of Chem., Bull. 137, 52.—The methods employed for sepg. mixed colors, and for the isolation of an oil-sol. color in a condition suitable for identification are given. Mention is made of the confusion of names and the difficulty of securing pure dyes in commerce, which has resulted in the many conflicting statements regarding their reactions published in the literature. An exact knowledge of the distribution ratios of the various acid dyes between amyl alc. and dilute acid for certain given concs. of acid in the water layer would be of great value in dealing with unknown mixts. A scheme is given whereby 12 oil-sol. colors in ether soln. may be sepd. by successive shakings with NH<sub>4</sub>OH, dil HCl and NaOH, and identified by means of the amines formed by reduction of the color with TiCl<sub>3</sub>.

A. F. Seeker.

Report on Spices. A. F. SEEKER. U. S. Dept. Agr., Bur. of Chem., Bull. 137, 80.—A detailed method for obtaining the ether extract and I no. of the extract of paprika. 2 samples contained 5% of olive oil, which were readily distinguished by the low I no. of the ether extract (109–115), pure paprikas having values above 125. The ether extract is obtained in a manner similar to the official method for alc. extract in spices, the chief precautions found necessary being care in selecting a pure solvent free of water and alc., and, when drying the extract, avoiding prolonged heating which causes oxidation of the oil and consequent lowering of the I no.

A. F. SEEKER.

Report on Vinegar. R. W. Balcom. U. S. Dept. Agr., Bur. of Chem., Bull. 137, 57.—The amt. of solids in 18 samples of vinegar was found to average 0.113 g. per 100 ce. less after 4 evapns. than by a single evapn. as in the official method. Titration of the residues showed that 0.059 g. of this was due to retention of AcOH by the solids. The greatest loss in solids occurs between the 1st and 2nd evapns., losses from subsequent evapns. being generally less than 0.05%. It is recommended that solids be detd. after 3 evapns., working upon the same amt. of material as in the case of wines. Reducing sugars may be detd. in vinegar by Munson and Walker's method without previous neutralization or clarification. A volatil non-sugar occurs in vinegar which makes reducing sugar results too high by 0.075-0.171 g. per 100 cc., and which can

be detd. in the distillate by reduction of Fehling's soln. Cooperative results on the detn. of pentosans and glycerol were satisfactory, but reports on color removed by fuller's earth varied widely, due probably to differences in the quality of the earth employed.

A. F. Seeker.

Determination of Glycerol in Vinegar and Characteristic Glycerol Ratios. S. H. Ross. U. S. Dept. Agr., Bur. of Chem., Bull. 137, 61.—The detn. is made by a modification of the official method for wines, all evapns. being conducted on a water bath maintained at  $85-90^{\circ}$ . Two evapns. are made to expel AcOH, the usual treatment with sand, (Ca(OH)<sub>2</sub>, EtOH and ether being then followed. The glycerol is finally purified with basic Pb acetate and Ag<sub>2</sub>CO<sub>2</sub> and detd. by oxidation with standard K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> as in Hehner's method. Genuin vinegars have a fairly constant EtOH and acid to glycerol ratio, which may be found by the formula R = [(a + 0.046) + (V + 0.06)]/(G + 0.0307), in which R =ratio of combined EtOH and acid to glycerol on equiv. basis, R =EtOH found, R =FtOH found, R =FtOH found, and R =FtOH found, the numerals being the factors required to reduce all these to a normal equiv. basis. The ratios on 3 vinegar products varied R =FtOH ratio of solids to glycerol (also believed to be of value) on the same samples varied was R =FtOH.

Problems of the Cider Maker. CROCHETELLE. Bull. soc. ind. Amiens, 48, 177-85.—The production of cider is exceedingly variable in France. The industry is not organized like that of brewing and the cider suffers in popularity as a drink because of the lack of a uniform product. Some of the processes which have been employed to obtain a uniform well regulated method of cider manuf. are as follows: Treating the juice with KHSO<sub>2</sub> to prevent fermentation. This gives a product of a good appearance which keeps well, but it has the taste of SO<sub>2</sub> and it has not been decided that SO<sub>2</sub> does not have a deleterious effect on those with weak stomachs. Washing the apples and all vessels with a weak formalin soln. gives good results but is contrary to the law; a similar treatment with Ca(ClO)<sub>2</sub> or Ca(MnO<sub>4</sub>)<sub>2</sub>, which processes are now being studied, as is the prevention of fermentation by pptg. the nutrient N compds. from the cider. One of the most promising methods of obtaining a constant supply of cider of uniform quality is to prepare it from dried apples by the diffusion process.

R. F. BACON.

Influence of Potassium Dichromate in the Analysis of Milk. LEON GARNIER. J. pharm. chim., 3, 55-9.—The author added 1 part of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> to 1000 parts of milk as required by the French law to arrest fermentation and analyzed the milk periodically for 3<sup>1</sup>/<sub>2</sub> months. Exam. of the results show that while the acidity, refractive index and cryoscopic figures increase slightly, the quantity of lactose diminishes a little. The variations are sufficiently limited however to enable one to apply a factor in correcting them.

V. K. Chesnut.

The Alleged Increase of Bacteria in Milk thru Mechanical Action. Ernst Gutzeit. Milchw. Zentr., 7, 193-211; thru Chem. Zentr., 1911, II, 44.—By filtering or centrifuging milk Dunbar and Kister (Milch. Ztg., 28, 787) obtained in certain expts. a lowering, in certain others an increase in the bacterial count; they explained the increase thus, that in raw milk often very many bacteria were united into a clump, from which but one colony grows on the gelatin plate. By filtering or centrifuging this clump is broken up, and there is hence shown an apparent increase in the organisms. Severin (Centr. Bakt. Parasitenk II, Abt., 14, 605; cf. also Severin and Budinoff, Ibid., 14, 463) obtained similar results but he ascribed the increase in the bacterial count to a stimulation of the multiplication processes of the bacteria. In G's. expts. filtration was made thru small pieces of porcelain in a specially constructed app. in such a manner that in 1 hr. 1 l. of filtered milk was obtained. To obtain solely the effect of the filtration and to exclude all other factors, such as the influence of air, the

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author compared the filtered milk not only with an unfiltered sample, but also with raw milk which at the same time was allowed to run thru the app. containing no porcelain. The result obtained by the author is that filtration is without influence in increasing the no. of bacteria. In repeating the centrifuging and shaking expts. of Severin, the author concludes that the increase in bacteria which occurs during the filtering, centrifuging or shaking of milk is not an apparent increase due to the breaking up of clumps of bacteria, but an actual increase, not to be explained as a stimulation of process of multiplication, but is simply a function of the time, temp. and other common factors influencing the growth of organisms. The fact that in correctly made tests, the filtration of milk causes an actual decrease in the no. of organisms was proved by Weigmann (Arb. Vers. Stat. Molkereiwesen zu Kiel, 1910, 57) using a Kröhnke's sand filter, in which the decrease averaged 17.5%.

Examination of Lactose and By-products of Lactose Manufacture. II. A. BURR AND F. M. BERBERICH. Chem. Ztg., 35, 776.—The tests of the purity of milk sugar based upon the following: (1) Mixt. of cheaper sugars or starches; (2) ash content from chalk, gypsum, etc.; (3) solubility in equal amt. of hot water without turbidity; (4) presence of heavy metals from careless mfg.; (5) N content; (6) free acid or base; (7) presence of Et<sub>2</sub>O-sol. material. These tests and their results are discussed, as is also the behavior with strong H<sub>2</sub>SO<sub>4</sub>. Tables of analyses are given and commented upon. III. 1bid., 794-6. The most important test with lactose is for the presence of other cheaper sugars. Many tests are known, of which the best is with conc. H<sub>2</sub>SO<sub>4</sub>. Adulteration is shown by the brownish yellow coloration so produced. Another test is the insolubility in alc., but this has been discarded from the last edition of the German Dispensatory. The relation between N content and bacterial contamination is discussed, as are other specific tests.

D. S. Pratt.

Milk as a Powder. F. MARRE. Rev. gen. chim., 14, 229-32.—A review of the various processes and patents for the evapn. and desiccation of milk. The method proposed by Fernando, Leconte and Lainville, in which the H<sub>2</sub>O is frozen instead of boiled out, is briefly described.

H. S. BAILEY.

What is Milk Chocolate Powder? CARL GREMERT. Z. offent. Chem., 17, 195-6.

—This is a protest by the Assn. of German Chocolate Migrs. against the recently adopted chocolate standards.

C. P. WILSON.

The Composition of Diabetic Foods. F. W. F. Arnaud. Pharm. J., 87, 176-9.

—Of 12 samples of gluten flour and bread representing 7 manufacturers, the products of only 1 firm were satisfactory; the starch content varied from 5.8-70.51% and proteins 8.12-86.19%. A 2 g. sample was dried at 100°, extracted with Et<sub>2</sub>O or petroleum ether to det. the fat, treated with EtOH 95% to remove sugar which was detd., and then extracted with H<sub>2</sub>O, the soln. being made up to 250 cc. and used for detg. the amt. sol. in H<sub>2</sub>O; including sol. ash, N and dextrin, the last is detd. by deducting the sum of the ash and protein from the amt. of dry residue; the starch was detd. in the residue insol. in H<sub>2</sub>O.

H. C. FULLER.

The Use of Colloidal Chemistry in Testing Foods. S. ROTHENFUSSER. Z. angew. Chem., 23, 977.—Certain sugars when in the presence of colloids have the property of being completely pptd. along with the colloids. This fact is used as the basis of a test for these sugars.

G. W. STRATTON.

The Primitive Fig and its Relation to the Capri Fig and the Domestic Fig. A. TSCHIRCH AND R. RAVASINI. Univ. Bern. Arch. farm. sper., 12, 49-54; cf. C. A., 5, 2301.

A. W. Dox.

The Value of some Tropical Grasses. A. EICHINGER. Pflanzer, 7, 26-32; Chem. Zentr., 1911, I, 1239.—Tables give the content of raw and pure protein, fat, N-free

extractives, raw fiber and ash of the various tropical grasses. The difference in the constituents and the feeding value of young, ripe, and old grasses is given.

M. X. SULLIVAN.

Water-soluble Carbohydrates in Chestnut Meal. GIOVANNI LEONCINI. Stas sper. agrar. ital., 44, 113-8; through Chem. Zentr., 1911, I, 1873.—To det. the water-sol. carbohydrates, proceed as follows: Shake 16.35 g. of the meal with 98 cc. water, add 2 cc. basic Pb acetate soln., and filter. Polarize the filtrate. In a number of samples, the author thus found an average of 26.25% sucrose, when the water content was 12.15%. The check with Fehling's soln. gave 26.71%. Contrary to reports of other authors, erythrodextrin, amylodextrin, and achroödextrin could either not be detected or detected only in traces. The presence of larger quantities of the dextrins was merely the result of too high a temp. during the roasting.

J. A. LECLERC.

Report on the Cultivation Experiment of the German Cultivation Stations in 1910. C. v. ECKENBRECKER. Z. Spiritusind., 1911, Erganzungsheft., 3-60.—The starch content of potatoes varies between 14.3 and 22.3%. Plots fertilized strongly with Chili saltpeter yielded more potatoes but these contained somewhat less starch than those with N-fertilizers. The protein was 1-2% higher so that N-fertilized potatoes are the most valuable (cf. also following abstract).

E. J. WITZEMANN.

Potato Analyses of the Harvest of 1909 and 1910. J. F. HOFFMANN. Z. Spiritusind., 1911, Ergānzungshejt., 61-4.—The exam. of various kinds of potatoes ought to
show some tubers, which because of remarkable properties such as a low water content
or high protein value, would be valuable for further cultivation. A 2 years' study did
not develop any notable examples of this kind. Harvest of 1909; protein content
1.27-3.02%, starch 1.06-24.88%. Harvest 1910; protein 1.47-2.43%, starch 14.1622.5%. Starch was detd. either by the tables of Foth from the density which was
detd. by means of the Reimann balance or by detg. the water and subtracting 5.75%
from the dry substance. The difference in results by the 2 methods was 3.5%.

E. J. WITZEMANN.

The Determination of Nicotine. G. BERTRAND AND JAVELLIER. anal., 16, 251-6.—Nicotine is one of the most valuable insecticides used in viticulture, hence tobaccos should be cultivated which contain a high % of this alkaloid. Silicotungstic acid ppts. nicotine in soln. of 1-1,000,000 in H<sub>2</sub>O, giving SiO<sub>2</sub>.12WO<sub>2</sub>.2H<sub>2</sub>O.-2C10H14N2.5H2O a pink cryst. compound, which at 120° loses 5 H2O and calcined gives a greenish residue of SiO2 and WO3. The alkaloid is extracted from the finely powdered tobacco (10 g.) with about 300 cc. b. 5% HCl using a reflux condenser. Filter, and in 250 cc. of the filtrate ppt. the nicotine with a 10-20% soln. of K silicotungstate. The ppt. after standing for a few hrs. is filtered and washed with dil. HCl. The ppt. is transferred to a flask with MgO and distilled with steam. The nicotine is titrated in the distillate with standard H<sub>2</sub>SO<sub>4</sub> using alizarin sulfonate as an indicator. In the presence of pyridine, the nicotine is again pptd. by silicotungstic acid, this ppt. decomposed by dil. Na<sub>2</sub>CO<sub>3</sub> soln. and the nicotine shaken out with CHCl<sub>3</sub> in which solvent it is detd. polarimetrically.  $\alpha$  for nicotine in CHCl<sub>2</sub> soln. (1-2% conc.) = R. F. BACON. 165.55°.

The Saponins (KOBERT). 17.

Absorption of Aluminium from Food (STEEL). 13.

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# 13. NUTRITION.

#### C. F. LANGWORTHY.

The Significance to the Digestion of Cellulose of Recent Investigations in the Field of Physiological Chemistry. Emil Abderhalden. Z. Oesterr. Ingenieur-Architekton-Vereins, 1911, 11 and 12.

E. J. Crane.

Nutrition with Simpler Products of Split Proteins. F. FRANK AND A. SCHITTEN-HELM. Erlanger med. klinik. *Munch. med. Wochschr.*, 58, 1288-90.—Protein that has been artificially digested till it no longer gives the biuret reaction, and given by mouth, is non-irritating and readily synthesized by the body. G. R. Henry.

The Time of Retention of Liquids in the Stomach. F. MEYER. Exp. biol. Abtg. kgl. path. Inst., Univ. Berlin. Z. physiol. Chem., 71, 466-71.—Tho the psychical factor may affect the early part of the motility curve, for the question of the total time that liquids take to pass the stomach it is negligible. It makes no difference whether liquid is given by the mouth, or by stomach tube or gastric fistula. Using dogs with duodenal fistula, the author found that H<sub>2</sub>O and physiol. NaCl soln. pass through the stomach in the same time (79-81 mins. in one expt.), while 2% NaCl and 2% sugar pass more rapidly, and 8% sugar much more slowly. The divergent results of Best and Cohnheim (Ibid., 69, 117) depend on differences in method.

A. HUNTER.

The Retention of Liquids in the Stomach. Fr. Best and Otto Cohnheim. Z. physiol. Chem., 72, 502-3.—A reply to Meyer (preceding abstr.). G. M. Meyer.

Glycogenic Properties of Dihydroxyacetone. St. Mostowski. Compt. rend., 152, 1276.—Dihydroxyacetone (30-60 g.) was fed to fowls which had been starved for 4-5 days and the livers of these fowls and of control animals were examined 7 hrs. later for glycogen. In each instance there was a considerable formation of glycogen. No glycogen was found in the muscles.

G. M. MEYER.

The Glycogenic Property of Glucosoamine. F. ROGOZIUSKI. Compt. rend., 153, 211-3.—Glucosoamine injected into chickens previously starved to free them of glycogen does not give rize to glycogen. The yeast cell is also incapable of converting glucosoamine to glycogen.

G. M. MEYER.

Protein Metabolism in Phlorhizin Diabetes. C. G. L. WOLF AND EMIL OSTERBERG. Cornell Univ. Med. Coll. Am. J. Physiol., 28, 71-80.—Fasting dogs were made completely diabetic with phlorhizin and the urins collected by catheterization. There was no apparent relation between acidosis and creatine; Taylor and Cathcart showed the same for dogs after moderate doses of phlorhizin (C. A., 5, 1954). The creatine increases during the poisoning to such an extent that the ratio of creatine N to total-N steadily rizes. A comparison of the creatine N, total N, and total S excreted indicates that the creatine is not the result of the catabolism of a particular kind of protein.

J. F. Lyman.

The Absorption of Aluminium from Aluminized Food. MATTHEW STEEL. Coll. P. and S., Columbia Univ. Am. J. Physiol., 28, 94-102.—Alum fed to dogs with Al-free food, or alum fed in biscuits made with alum baking powder was afterwards detected in the blood. The Al showed no tendency to accumulate in the blood. AlCla injected intravenously passed into the feces during 3 days to the extent of 5.55-11.11%.

J. F. LYMAN.

The Effect of Parathyroidectomy upon Metabolism. ISIDOR GREENWALD. Coll. P. and S., Columbia Univ. Am. J. Physiol., 28, 103-32.—The excretion of NH, after parathyroidectomy was increased slightly if at all. The elimination of N compds. of unknown nature was increased pointing to a diminished activity of the liver. The

results do not support the view that parathyroid tetany is due to intoxication with NH<sub>2</sub> or carbamic acid. The P in the urin was markedly reduced after the operations, and increased after tetany set in. Total S was increased, the increase being chiefly in the forms of inorganic and neutral S. Creatine was much increased. Total N increased only after the appearance of tetany. The blood during tetany contains the normal amt. only of NH<sub>2</sub>.

J. F. LYMAN.

The Eradication of Beri-beri from the Philippine (Native) Scouts by means of a Simple Change in their Dietary. W. P. Chamberlain. Philipp. J. Sci., 6, 133-46.

—The true factor for the elimination of the disease among the native scouts is regarded by the Board for the study of Tropical Diseases as the reduction in the rice consumed and the substitution of a legume. The board favors the polished rice theory of beriberi production. The adoption of an undermilled grain will allow rice to be used more freely. Unpolished rice can be purchased in Manila containing 0.52% P<sub>2</sub>O<sub>3</sub> while the polished variety contains only 0.26% P<sub>2</sub>O<sub>3</sub>. Rices containing over 0.4% P<sub>2</sub>O<sub>3</sub> apparently prevent beri-beri.

Changes in Protein Metabolism in some Maladies. Gerolamo Cuneo. Irrenanstalt, Genua. Bull. sci. med., 81, 565-603; thru Zentr. Biochem. Biophys., 11, 833.—Continuing earlier work in which the results indicated that neuralgia of the trigeminus is caused by changes in protein metabolism causing autointoxication, it is found that protein metabolism is abnormal in depressive maniac psychosis and in dementia praecox. The protein metabolic and catabolic products are not eliminated normally. The more complex protein cleavage products are present and produce the symptoms. In maniac depressive psychosis the cause is abnormal hydrolysis and de-amination in the intestins while in dementia praecox certain glands which normally produce internal secretions of antitoxic and protective actions fail to function properly and thus allow toxic substances to circulate.

I. K. Phelps.

# 14. WATER, SEWAGE AND SANITATION.

#### EDWARD BARTOW.

Effect of Salts in Potable Water. Anon. Engineering, 92, 134.—The article is the result of 15 years' unsuccessful litigation on the part of the city of Magdeburg, Saxony, with the National Copper Mines and numerous salt mines on the River Elbe from which the city draws its supply, and considers in more or less detail the contamination of the German rivers with refuse from such mines. MgCl<sub>2</sub> previously has been considered harmful, but waters containing 100 to 122 mg. per l. are being used as drinking supplies without apparent harmful results. The writer concludes "the water source need not necessarily be condemned outright on account of a relatively high percentage of salts."

Permission Granted in New York for "Reasonable" Contamination of a Village Water Supply. Eng. Record, 64, 255.—A recent decision of the State Court of Appeals in George vs. Village of Chester, permits the "reasonable" use by riparian owners of a body of water used for municipal supply. Nuisances are prohibited.

LANGDON PEARSE.

Reinerz Bath in Schlesien and its New Healing Springs. R. Woy. Z. offent. Chem., 10, 181-92.—A historical sketch is given of the principal baths with reference to the changes in the chem. comp. Notes on the temp. changes are included. The results are given of the chem. and phys. investigation, carried on in the summer of 1910, of the waters from 4 springs. Included in the detns. are: the sp. cond., lowering of the f. p. and the radioactivity. The water from all 4 springs is radioactive. The

chem. comp. of the waters from the Reinerz springs is compared in tabular form with the comp. of waters from a number of other springs.

C. P. WILSON.

The Relation of Microscopic Plankton to the Chemistry of Waters. KOLKWITZ, Mitt. kgl. Prajungsamt Wasserversorgung, Abt., 14, 144-215 (1911); Chem. Zentr., 1911, 1440.—The author found during his investigation, at times more algae than bacteria per cc. present in waters. To judge by some observations, a certain bacterial flora seems to be accompanied by definit species of plankton. 200 samples were examined as to their plankton content, and the species found in each sample were tabulated. Inasmuch as the plankton are present in sufficiently large numbers in surface waters I cc. can well serve as a unit as it does for bacterial investigations. The plankton originating from the Elbe, Havel, Spree, Rhine, Mosel, Main, Weser, Oder, Weichsel, from lakes in North Italy and Switzerland, and from the ocean (East and North Sea) are enumerated alphabetically. The author designates as polysaprobic the group of plankton which to the greatest extent flourish in sewage contaminated waters containing peptone and like compds. This group tends to improve the putrescible character of the water to some extent, together with the  $\alpha$  mesaprobic organisms. The latter group is principally represented in waters which undergo active self-purification. The plant and animal organisms which exist in waters more or less similar in comp. to purified drain waters are called  $\beta$ -mesaprobic. One can easily recognize the chem, character of a water, as well as its state of self-purification, with a table showing the number of the organisms of these different groups per cc. in alphabetical order.

ARTHUR LEDERER.

Denitrifying Bacteria of Percolating Beds. Lemoigne. Compt. rend., 152, 1873-5.—Denitrification usually is considered the result of defective aeration of biological filters (cf. W. Mair, C. A., 3, 1196). Maze demonstrated (cf. Ann. inst. Pasteur, April, 1911) that denitrification is a regular step in the assimilation of N. Tests were made to det. the role of the denitrifying bacteria in well aerated beds. The common bacteria have been isolated, altho many break down nitrates. Two species are particularly prevalent, bacillus  $\alpha$ , perhaps B. subtilis; bacillus  $\beta$ , very like B. subtilis, in red colonies. These two changed nitrates to NH<sub>2</sub> direct. Tests in different shaped vessels with a bouillon containing 1% NaNO<sub>3</sub> showed that aeration favors the denitrification with bacteria which can not utilize the O of the nitrate. Apparently nitrification and denitrification occur simultaneously. The bacteria resembling B. subtilis are very abundant in filters. Other denitrifiers, however, do exist, which may be found by later expts.

The Clarification of Surface Waters with Alum Previous to Slow Sand Filtration. G. NACHTIGALL AND L. SCHWARTZ. Ges. Ing., 34, 545-56.—Filter expts. show that it does not pay to add more alum to raw water than is required to obtain a good flock in the time consistent with the practical operation. Of course when alum is used in larger quantities the flock is better, and consequently more bacteria, and suspended coloring matter will settle to the bottom, but the improvement of the raw water thus gained is not in proportion to the higher cost of alum, and furthermore one can hardly expect an increase in the length of the operation of the filter. In the sand filtration it is advantageous to add to the raw water a quantity of alum just sufficient to produce the maximum flock in the time allowed for operating purposes. The quantity of alum to be added can be detd. experimentally by adding various quantities of a 1% alum soln. to a series of 1 gal. samples of the raw water. The temp. should coincide with the temp. to which the filters are exposed. The time element must also coincide with the practical scale of operation. The efficiency of the coagulation can be roughly measured by visual inspection. Practically it is impossible to prevent the deposit of small quantities of the flock on the sand filter. These small quantities may, under some circumstances diminish the efficiency of the filter. From a hygienic standpoint, however, the coating results in a better effluent. ARTHUR LEDERER.

The Carbon Dioxide Content of Various Forms of Natural and Artificial Carbonic Acid Baths, and of the Atmosphere above these Baths. R. Von der Heide. Z. Balg. Klim. Kurorthyg., 3, 644-58; Chem. Zentr., 1911, I 1245.—The expts. were carried on at the bath of Altheide and Ems. At the Altheide bath the largest quantity of CO<sub>2</sub> could be found at the bottom of the water in the bath tub, while at Ems the largest quantity was present in the surface layer. The explanation lies in the different prep. of these waters, and in the mixing of the CO<sub>2</sub> water with the water which is added to regulate the temp. Artificial baths gave off more CO<sub>2</sub> than natural baths. The bath at Ems, at temp. of 28°, 25° and 20°, at the beginning and at the end contains more CO<sub>3</sub> than the bath at Altheide. The increase of CO<sub>3</sub> at a decreasing temp. is more pronounced at the Ems than at the Altheide bath.

Arthur Lederer.

Laboratory Experiments with Permutite for the Softening of Water. H. G. Anders. Wochschr. Brau., 28, 78-80; Chem. Zentr., 1911, I, 846.—The expts. show that permutite is applicable for the softening of feed-waters, but not for mash-waters because it takes up Na salts. A permutite filter may be completely regenerated by use of NaCl soln.

E. J. Crane.

Coagulant Control. Monessen Filters. Eng. Record, 64, 282.—The coagulant feed is regulated by a large float in a chamber containing an orifice thru which the entire pumpage flows. The float actuates an overflow weir in the coagulant orifice boxes. A proportionate flow is thus obtained.

LANGDON PEARSE.

Some Notes on Locomotive Water Supply. L. D. GRIMES. Purdue Eng. Rev., 1911, 118-21.—Practice of the Union Pacific Ry. in locating and maintaining water supplies.

E. Barrow.

The Use of Hypochlorite in Water and Sewage Purification. J. A. NEWLANDS. Proc. Conn. Soc. of Eng., 1911, 87.—A review. E. BARTOW.

Corrosion of the 350-Mile Thirty-inch Coolgardie Steel Pipe-line in Western Australia. Anon. Eng. News, 66, 221-5.—The receiving and service reservoirs of this supply main afford very little reserve capacity, thus making the condition of the pipe line of unusual importance. The materials used, their construction, and a general description of the protective coatings used are considered at length. External pitting was first noticed a little over 2 years after the line was put in service; in 3 years 151 This corrosion was worst in low, wet, clayey places. Recoating of bad sections, exposure of pipe, and draining or cutting away of surrounding soil greatly improved conditions. The action of sol. salts in the soil on metal pipe was carefully investigated. After 5 yrs. of service, reduction of 12-53% in carrying capacity resulted in much tuberculation and internal corrosion. The tubercles were 1/4-1 1/4 in. in height, and the pits underneath rarely exceeded 1/8 in. in depth. The protective coating was virtually destroyed in the worst sections. The trouble was laid to the water which contained 13.3 grains per Imp. gal. NaCl, 2.3-2.98 grains of MgCl, with only about 2 degrees of protective hardness. G. F. Deacon, Sir Wm. Ramsey and Otto Hebner held that dissolved O was the cause of the corrosion and recommended deaeration and addition of lime. The local board attributed the trouble entirely to electrolysis, and recommended lime, some changes in piping, and storage to aid exclusion of air. In one yr., lime has reduced the corrosion and a coating of CaCO, is forming on the interior of the pipe.

Mitigation of Electrolysis of Pittsburgh Water Pipes. E. E. LANPHER AND L. B. SMITH. Eng. News, 66, 231.—Responsibility for destruction of water mains was fixed on the Philadelphia Co. which operates all the electric railways in the vicinity. In-

vestigation showed several centers of serious trouble adjacent to the various power houses. A max. positive potential of 24 v. was found where the trouble was greatest, varying to 5 v. (positive). Cu bonding of every calked joint and the installation of a "return pipe feeder system" were recommended. 1,000,000 c. m. cables run from the power house to and along the pipes in danger, in the worst districts, reduced potential to about 0.5 v. (oscillating + and —). The deterioration is now almost negligible.

D. K. FRENCH.

Bangor Waterworks Notes. Munic. J. Eng., 31, 263-4.—A summary is given of the 1910 operation of an old filter plant consisting of 24 Warren filters and 2 coagulant basins with combined capacity of 1 1/2 mill. gals. 1.43 grains per gal. of Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> were used. Specifications for the same required over 17.5% Al<sub>2</sub>O<sub>3</sub>, and not to exceed 37.5% acid, 0.4% iron, 47% water and 0.25% insol. matter. The payment was specified on a sliding scale on the basis of the Al<sub>2</sub>O<sub>3</sub> and Fe present. During 2 1/2 months Ca(OCl)<sub>2</sub> was used to increase the bacterial efficiency. 0.25 of a grain per gal. of coagulant was saved thereby.

LANGDON PEARSE.

The Municipal Water Purification Plant of Montreal. Eng. Record, 64, 280-1.

—This \$1,500,000 plant has a capacity of 60 mil. U. S. gals. daily. The works comprize a pumping plant, 16 prefilters of the rapid filter type, 16 final filters (slow-sand type), a clear water reservoir, and a concrete wash-water tower. 12 in. assorted gravel is overlaid in the final filters by 27 in. of sand, 10% of which shall pass a 50 mesh-sieve, not more than 10% shall pass a 60-mesh sieve, and 90% must be less than 2.1 mm. size. The 16 units are each 340 × 55 ft. inside.

New Cincinnati Water Works. J. S. GETTRUST. Purdue Eng. Rev., 1911, 139-44.
—During the 3 yrs. following the installation of filters the typhoid case rate of Cincinnati has been reduced 83% and the death rate 70% over the 3 preceding yrs.

An av. of 1.85 g. of CaO was used during the yr. The temporary hardness was increased from 46.2 to 49.0; the total hardness 74.4-85.7. The bacteria were decreased from 9300 to 75 and the turbidity from 225 to 0.

E. Bartow.

Water Supply of Cleveland, Ohio. Report Water Works Division, 1910.—The daily bacterial av. was 272 per cc., a larger number than was found during 1909. B. coli was also present more frequently. The turbidity averaged 17.06 against 28 for 1909 and 30.64 for the yrs. 1905-8.

The Oxidization of Sewage Polluted Salt Water. Col. W. M. Black and Prof. E. B. Phelps. Munic. J. Engr., 31, 199-202.—In a report to the Board of Estimate of N. Y. City, the conclusions given are that the best criterion of the purity of water in N. Y. harbor is the dissolved O, and that this should not be drawn below 70% of the full satn. value. The principal difficulty is local pollution. Septic treatment, followed by forced aeration, is recommended. This process is peculiarly adapted to cope with summer conditions. If winter conditions demand treatment at all, then the summer conditions can not be met by aeration. Absorption of O from the air will give an increase of 1.9% of the satn. value in 24 hrs. A table is given of the amt. of O absorbed by a quiescent body of fresh water at 20° C. at stated times and depths. Forced aeration by blowing air thru the liquid, with a period of contact from 1.4 to 5.6 hrs. showed an improvement from 45 to 320%. From 0.09 to 0.77 cu. ft. of air was used per gal. of liquid. Crude, settled and septic sewages were experimented upon.

Tests of Sewage Spraying Devices. C. A. Hammond. Munic. J. Eng., 31, 232-4.

—An account of tests on nozzles of the Columbus and Taylor type, as well as a Columbus type with a single arm, and a glass deflecting cone with rounded bottom, for which a high efficiency is claimed.

LANGDON PEARSE.

Experimental Sewage Purification at Worcester. Matthew Gault. Eng. Record, 64, 242-3; Munic. J. Eng., 31, 257-60.—This summarizes the results of tests on 3 filters, one 5 feet deep, 0.028 acre in area, of 3/4 to 2 1/2 in. stone; one 7 1/2 feet deep, 0.014 acre in area of 3/4 to 2 1/2 in. stone. The first 2 years septic sewage, the latter 2 years sewage settled 8 hours, was applied. At a rate of 3 to 5 million gallons daily per acre clogging ensued in a year. Fresh sewage could be applied at a rate of 500,000 to 1,000,000 gals. per acre daily on 5 to 7 1/2 ft. beds. The septic treatment produced odors and suspended matter to clog both nozzles and beds. The 7 1/2 ft. filters produced better and more consistent results. Tables and diagrams are given of results of analyses.

LANGDON PRARSE.

The Flushing System for the Gowanus Canal, Brooklyn, N. Y. Eng. Record, 64, 212-4.—The Gowanus Canal is an old creek channel about two miles long, used for dock purposes. Since 1880 it has been a live nuisance. Dilution by flushing was proposed years ago. The soln. has been the removal of the foul water by a screw pump discharging thru a tunnel into Buttermilk channel. By this the settling suspended matter is removed. The current is toward the head end, favoring shipping, and the outlet may discharge above ebbtide freely without danger of losing suction. L. P.

Sewage Purification at the Mont Meely Works, Paris. M. MATHIEU. Annales des. Ponts et Chausses., 47, No. 5; Eng. Record, 64, 218-20.—In the sanitary district of the river Seine are included 77 towns besides Paris. Investigations showed that sewage farming was impracticable. In 1905 a project was started to treat 2.85 million gal. daily from Ivry and Vitry (population 45,000), 2.2 million to be handled on double contact beds, and 0.63 million on percolating filters. The sewage is pumped 5.6 miles to Mont Mesly. A grit chamber followed by 11 septic tanks was designed, with 24 hrs. period of flow. The contact beds, however, were not built, but percolating filters were, of total cap. 5.55 million gals. per day. On 90,420 sq. ft. distribution is by a siphon device, on 45,210 sq. ft. by a fixed nozzle, on 90,420 sq. ft. by a traveling distributor. The beds are 6 1/2 ft. deep. Crushed stone and slag are under trial, both graded and of uniform size. The effluent is settled 2 hrs. before discharge into the Seine. The sludge is dried on sand beds and distributed to farms. The filters sprayed by nozzles have given the best results.

The Proposed Disinfection of Sewage at New Bedford. W. T. SEDGWICK AND E. B. PHELPS. Eng. Record, 64, 269-70.—Disinfection during 98-99% of the yr. is advizable. From 100 to 150 lbs. of Ca(OCl), is required per mil. gals. Phelps recommends screening as a preliminary treatment.

LANGDON PEARSE.

The Effect of Antimony in Experimental Trypanosomal Infection. MORGENROTH AND ROSENTHAL. Berl. klin. Wochschr., [2] 48, 61; Chem. Zentr., 1911, I, 834.— The delitescence of this organism is speedily effected by as little as 0.2 cc. of a 0.1% soln. of tartarized Sb. By continued treatment with tartarized Sb resistance of the Trypanosoma to its effect was not developed. K hexatantolate neither influences trypanosomal infection nor has any prophylactic value, but rather protects the Trypanosoma against the action of Sb and arsenacetin.

E. J. Crane.

Determination of Arsenic and Iron Salts in Mineral Waters (AGENO, GUICCIARDINI). 7.

Pathogenic Properties of Gases Discharged by the Exhaust Pipe of Gas Engins (DELÉPINE). 21.

Ger., 235,752, July 2, 1909. CHEM. FABR. GRIESHEIM-ELEKTRON, Frankfurt

a/M. In the manuf. of concentrated formic acid from formates by decomp. with acids, organic sulfonic acids are employed to liberate the HCOOH. The crude sulfonating mixt. obtained in the production of sulfonic acids is applicable directly. The resulting excess H<sub>2</sub>SO<sub>4</sub> does not decompose the HCOOH as it is dild. by means of the sulfonic acid.

# 15. SOILS AND FERTILIZERS.

#### F. P. VEITCH.

The Distribution of Organic Constituents in Soils. Oswald Schreiner and E. C. Lathrop. J. Frank. Inst., 172, 145-51.—Pentosan was found to be a general constituent of soil organic matter as was also pentose sugars or pentose-containing complexes. Histidine was found in 17 out of 24 soils; arginine, in only 2; cytosine, in 10 out of 23; xanthine, in 5 out of 24; hypoxanthine in 9. Some of the soils contained xanthine only, some hypoxanthine only and some both. Dihydroxystearic acid was found in 1/3 of all soils examined, generally accompanied by xanthine or hypoxanthine or both. It is harmful to plants. Among the good soils only 2 contained dihydroxystearic acid while 51% of the poor soils contained it.

M. X. Sullivan.

Dihydroxystearic Acid in Good and Poor Soils. O. SCHREINER AND E. C. LATHROP. J. Am. Chem. Soc., 33, 1412-7; cf. preceding abstr.—A total of 84 soil samples from 18 states were examined for dihydroxystearic acid; of these 24 were subsoils. Of these soils 33% showed its presence. It was contained in soils under long cultivation, as well as in virgin lands, in soils continually cropped as well as in soils under permanent sod. It is likely to be found in soil work anywhere. It is either a direct or an indirect factor in low productivity, direct because of its harmful effect and indirect as arizing and accumulating under poor soil conditions.

M. X. Sullivan.

The Action of Wax Solvents and the Presence of Thermolabil Bacteriotoxins in the Soil. R. Greig-Smith. Proc. Linnean Soc., 35, 808-22; thru Zentr. Biochem. Bio-phys., 11, 852-3.—H<sub>2</sub>O extracts of soil contain a poisonous substance produced by the cacteria present. This product disappears on standing, is destroyed by sunlight and by heat, and retards plant growth. Its H<sub>2</sub>O soln. decomposes quickly. The soil particles are covered with a wax layer partly non-saponifiable and partly saponifiable in nature. A good distribution of this "agricer" is favorable to the soil as was shown by the higher production of toxins after the extraction of the wax. I. K. Phelps.

A Heretofore Unnoted Benefit from the Growth of Legumes. T. L. Lyon and J. A. BIZZELL. Cornell Univ., Agr. Exp. Sta., Bull. 204.—Timothy grown with alfalfa and cut early contained 51 lbs. more protein per ton of hay than timothy grown alone. Late-cut timothy contained 12 lbs. more of protein per ton when grown with alfalfa. Timothy with red clover gave 133 lbs. more of protein to the ton than grown alone. Oats grown with peas contain more protein than oats grown alone, and the yield of mixed oats and peas when cut for hay was 1050 lbs. greater per acre than the yield of oats alone on adjacent plots. Soil which had grown alfalfa 5 yrs. and from which the crop was not removed July 6, gave NO, averaging 4.4 pts. per mil. for each of the first 4 ft. of soil, but only 2.0 pts. per mil. with timothy grown 5 yrs. Soil which was kept bare until July 6, gave 15.4 per mil. of NO, when the previous croo (for 5 yrs.) was alfalfa, and 8.8 pts. when the previous crop was timothy. The rate of nitrification of (NH<sub>4</sub>), SO<sub>4</sub> was greater in alfalfa soil than in timothy soil whether limed or unlimed. The limit of nitrification was nearly the same in each soil. Liming about doubles the nitrification in 10 days and gives 2.5 times as much NO, in 20 days for each soil. Liming increases the yield of mixed hay by 37%; the % of alfalfa in the mixed hay is increased from 61 to 71; the % of protein in the alfalfa from 15.88 to 20.63; the % of protein in *Erigeron annuus*, a weed growing with the hay, is increased from 8.79 on the unlimed to 10.67 on the limed; and the nitrates in dry soil in pts. per mil. are increased from 4.3 to 8.1.

L. W. RIGGS.

What is White and Black Alkali? E. W. HILGARD. Science, 34, 21-2.—Black alkali is Na<sub>2</sub>CO<sub>2</sub> which after dissolving the humus of the soil leaves black spots on the land where the soln. has evapd.; white alkali leaves only the white crust of sulfate and chloride of Na. The sulfate is 4 times less injurious to vegetation than the carbonate while common salt stands in between in this respect. It makes little difference whether the Na carbonate is in form of bicarbonate, sesquicarbonate or monocarbonate; ultimately the effect is that of black alkali. Therefore, water containing considerable NaHCO<sub>2</sub> should not be used for irrigation.

M. X. SULLIVAN.

Rocks and Waters of the Roman Land with Respect to Lime. G. DE ANGELIS D'OSSAT. Atti accad. Lincei, 20, I, 349-54.—Description of a calcareous material found in the via Tiburtina under a thin layer of pozzolana and over lithoidal tufa. The capacity to absorb H<sub>2</sub>O and the velocity with which the H<sub>2</sub>O will rize to the top and evap. in the air were detd. Taking the annual rain fall at Rome as 901.8 mm., layers of this material 0.678-0.940 m. thick are sufficient to retain all the H<sub>2</sub>O without allowing any thru into subterranean circulation.

C. A. ROUILLER.

The Significance of Lead Arsenate Composition. W. H. Volck. Science, 33, 866-70.—Pb arsenate is often injurious to vegetation. Harmless arsenates contain Pb oxide and As oxide in the correct proportion to produce orthoarsenate Pb<sub>2</sub>(As<sub>2</sub>O<sub>4</sub>)<sub>2</sub>. Com. Pb arsenate consists of Pb<sub>2</sub>As<sub>2</sub>O<sub>7</sub> and PbHAsO<sub>4</sub>. When used as a spray the lead arsenate is subjected to neutral and alk. conditions and orthoarsenate is formed with the liberation of As oxide and sol. arsenates. The sol. As compds. do the injury. Samples which when boiled with NH<sub>4</sub>OH give a filtrate which gives a ppt. with Pb(OAc)<sub>2</sub> and AcOH, have excess of As and are as a rule injurious to vegetation. Samples which show no NH<sub>3</sub> test are practically harmless. Addition of Pb(OAc)<sub>2</sub> to the com. Pb arsenate spray until the water gives a reaction for sol. Pb was found beneficial in checking the injurious effect of the spray.

Lime and Humus on Soils. W. Tharr. J. Landw., 59, 9-57, 108-39.—It is well known that lime effects colloidal humus. By detg. permeability to water, it was found that humus colloid is similar in action to other colloids of the soil. Lime decreased plasticity of clays in the presence of humus, but increased it in the absence of humus. Humus and colloidal clay had the same effect upon the tilth. The shrinkage of soil is influenced by both. On liming a large part of K-zeolite goes into soln. Lime ppts. the soil colloids and lessens surface, thereby reducing the absorption of the soil. Absorption of soil is increased by lime if the bases set free are taken up by plants, or, if new absorptive substances are formed. It is lessened when the surface of the colloid is decreased, and not changed when the bases set free are not removed. Lime hinders or prevents crusting of soil. Humus has an absorptive effect and increases that of sand. Other results could not be classified.

R. O. E. Dayis.

The Availability of the Insoluble Nitrogen in Certain Commercial Fertilizers. B. L. Hartwell and F. R. Pember. J. Ind. Eng. Chem., 3, 584-6.—Using oats and millet as test plants the availability of the water-insol. N ingredients of com. fertilizers was detd. This availability was, with a few exceptions, practically equal to that in dried blood and N as tested by plant growth was in fair agreement with the availability as tested by the alk. KMnO<sub>4</sub> method.

M. X. Sullivan.

Determination of Nicotine (BERTRAND, JAVELLIER). 12. Sodium Chloride Fertilizer for Beets (STROHMER, FALLADA). 28.

# 16. FERMENTED AND DISTILLED LIQUORS.

#### ROBERT WAHL.

Researches on the Fluorides in Wine. P. Carles. Ann. chim. anal., 16, 296-9.

—The fluorides like the phosphates and other chemical compds. :hould be adjudged as medicaments when present in wine. These substances cannot play a useful role except in certain definit diseases. As a rule medicin should not be taken together with foodstuffs of any kind, unless in certain rare cases such a combination should prove to be of therapeutic advantage.

C. A. Nowak.

The Detection of Saccharose in Wines According to Rothenfusser. F. Schaffer AND E. PHILIPPE. Mitt. Lebenm. Hyg., 1, 303-11; thru Chem. Zentr., 1911, I, 1256. -The method of Rothenfusser (C. A., 4, 1524) for the detection of saccharose cannot be used to distinguish between gallized wines and genuin pure wines. This is because the blue color formed with diphenylamine is not specific for saccharose, and because in forming this color the carbohydrate and related substances contained in wine take the principal part, either directly or indirectly. The real cause of the color reaction shown by von Ekenstein and Blanksma (C. A., 4, 3222) to be the hydroxymethyl furfural formed from the hexoses; it is also recognized as the agent of Fiehes, the sesame oil, the  $\beta$ -naphthol and of the Liebermann's reactions. Ketohexoses give the reaction more easily than aldohexoses. This explains why the author has obtained the reaction both in wines containing cane sugar and also in fresh grape juice in which it is generally assumed there is no saccharose. The cases of fermented wines, in which the presence of saccharose is excluded and which will still give the blue color with the diphenylamine reagent are not so easily explained. R. F. BACON.

The Preparation and Composition of Madeira Wine. FERREIRA DA SILVA. Ann. Jals., 4, 57-65; thru Chem. Zentr., 1911, I, 1605.—The author reports briefly on the ordinary method of wine mfg. in Madeira and then gives a series of analyses of different Madeira wines, 60 of them by himself. His conclusions are as follows: The av. EtOH content is 20%, max. 23.30%, minimum 16.60%. Extract: mean 78, max. 178, min. 31.6 g. After subtracting the unfermented or added reducing sugars: mean 30, max. 82, min. 12.7 g. The tannin content according to the Zn(OAc), method is very small; the highest was 1.05, mean 0.15 and the lowest 0.07. The total acidity of these fortified wines is in general high, the mean being 5 g. per l. The sum, alcohol + acid, is as a mean 25, max. 29, min. 20. These values are only given as extraordinary and should not be compared to those found in ordinary wines. The Madeira wines are not dry but contain a greater or less amt. of uninverted sugar. Tannin is found in all Madeira wines.

A New Method of Handling Wine. PHILLIPE MALVEZIN. Bull. assoc. chim. sucr. dis., 28, 770-4.—When new red wine is allowed to stand in an atmosphere of N the color is brightened and it soon takes on an appearance and properties of an old wine. The intensity of color is reduced by the pptn. of tannin, the color material being carried down by N. Fermentation in the wine is interrupted in a short time by the introduction of N and aerobic bacteria are destroyed.

A. H. BRYAN.

Note on Lorraine Wine and the Calculation of Dilution. P. GRELOT. J. pharm. chim., 3, 97-100.—The full analytical data on genuin samples show a comparatively low ash content with a relatively high extract. Although genuin, they should be judged only by the standards for the weaker wines.

V. K. CHESNUT.

The Detection of Fusel Oil in Alcoholic Drinks. EDUARD HERZOG. K. k. Unters. Anstalt f. Lebensm. Czernourtz. Z. Nahr. Genussm., 21, 280-2.—The fusel oil test proposed by Hollander (C. A., 4, 781) is a furfural, not an amyl alc., reaction. The

reaction is sharper when  $H_8PO_4$  is used in place of HCl. By this reaction 0.3 mg. furfural can be detected in 100 g. 50% EtOH. The aniline acetate and  $\alpha$ -naphthol methods for detecting furfural are much more sensitive than Holländer's method. All reactions for the detection of fusel oil which are really furfural reactions are objectionable, as many other substances added to alc. drinks give either similar reactions or mask the characteristic color reaction of furfural.

R. F. BACON.

The Determination of the Percentage of Alcohol without Distillation and the Application of the Formulas of Blarez and Ordonneau. Bonis. Ann. jals., 4, 276-8; Ann. chim. anal., 16, 258-60.—The author states that the formula of Blarez is based on a large no. of detns. of actual alc. liquors, while that of Ordonneau is deduced theoretically for aq. solns. of pure sugar and alc. In most cases where exactness is required neither formula can be used, but the % of EtOH must be detd. by the method of distillation.

R. F. BACON.

Brewing and Modern Science. ADRIAN J. BROWN. J. Roy. Soc. Arts, 59, 939-50; 959-70; 975-82; 993-1003. J. M.

Contributions to the Method of Beer Analysis According to Balling. Doesness. Z. ges. Brauw., 34, 369-73, 385-90.—As a result of his investigations, the author comes to the conclusion that: 1. The number 2.0665 is too high, giving in the stored beer 0.15-0.20 more than the corrected p-value detd. previous to fermentation. 2. The yeast factor of Balling, 0.11, is likewise high; taking into consideration the amt. of alc. formed during the primary and secondary fermentation the same amounted to approximately 0.055 in one brewery. 3. If the p-value be detd. previous to fermentation the same must be corrected for the amt. of water introduced into the wort with the pitching yeast. 4. The smaller, less accurate formula for p gives in vat beer, owing to the poor binding of the  $CO_2$ , a higher value than the longer formula with the number 2.0665. With stored beers and correspondingly detd. m-value both formulas give the same results.

The Significance of Phosphoric Acid and its Combinations in the Brewing of Beer. F. Fernbach. Ann. Brasserie Distillerie, 14, 217-21; Z. ges Brauw., 34, 359-62.—The ash of barley amounting on the av. to 2.6% of the wt. of the corn consists chiefly of H<sub>2</sub>PO<sub>4</sub> which calc. to the K salt amts. to about 55% of the ash. The author believes that the H<sub>2</sub>PO<sub>4</sub> combinations play an equally important part in those changes whichare of greatest interest in brewing as do the fermentable sugars.

C. A. Nowak.

Contributions to the Method of Acid Determinations in Malt Worts and Beer. Ed. Moupang. Wockschr. Brau., 28, 329-31.—The results obtained may be summarized as follows: (1) Colorless phenolph. is the best adapted indicator; 1-2 drops are used for every 10 cc. of titrating soln.; (2) the acid detn. is easily carried out provided a sufficient diln. is worked with, 1: 10 for dark worts, 1: 5 for light worts; (3) the amt. of acid is best calc. to 1 hectaliter of 10% wort, and expressed as heretofore in g. of lactic acid; (4) in using 1/18 N Ba(OH), soln. for titrating the number of cc. required for 50 cc. wort (diln. 1: 10) multiplied by 100 gives directly the number of g. lactic acid contained in 1 hectaliter; It is only necessary to figure this value over to 10% wort. (5) with dark worts it is advizable to use 35 cc. of a diln. of 1: 10 for titration to obtain a sharper end reaction; (6) the use of asbestos for clarifying turbid worts, may cause variations from the true acid values; (7) the limit of error may be set at about 5 units.

C. A. Nowak.

The Necessity of Constructing a New Saccharimeter for Use in the Brewery. O. Mohr. Wochschr. Brau., 28, 304-6.—The expts. made lead to the following conclusions: (1) The construction of a new saccharimeter is necessary because, owing to the different surface tensions of cane sugar solns. and of wort the instruments stand-

ardized with cane sugar indicate a too high % of ext. in wort; (2) the scale of the new instrument should be graduated in % of ext.. not in degrees Balling; (3) the introduction of the wort saccharimeter also necessitates the exclusion of the Balling table from malt analysis. During the time of transition from one system to the other the % of ext. is to be figured according to Balling as well as according to the table constructed by the Standard Graduating Commission.

C. A. Nowak.

The Solubility of Carbon Dioxide in Beer. A. FINDLAY AND B. SHEN. J. Chem. Soc., 99, 1313-20.—Previous expts. on the adsorption of CO<sub>2</sub> by beer have shown that beer dissolves more CO<sub>2</sub> than the corresponding water-alc. mixt., which is attributed by Emslander and Freundlich to adsorption of the colloids in beer. According to the author's expts. CO<sub>2</sub> is less sol. in beer than in the corresponding water-alc. mixt.; which is contrary to Langer and Schultze. These investigators must have worked with beer supersat. with CO<sub>2</sub>.

C. A. Nowak.

The Alcohol Industry of the Philippine Islands. I. A study of Some Palms of Commercial Importance with Special Reference to the Saps and their Uses. H. GIBBS. Bureau of Science, Manila, P. I. Philipp. I. Sci., 6, 99-143.—Of the total amt. of alc. and alc. beverages produced during the last fiscal year 93% was distilled from the saps exuding from palm trees. About 22 different beverages (mostly imitations of the well known brands) are manufactured from the rectified alc. The most popular of these, anisette, anisado, gin, vino de coco. and vino de nipa, vary between 10 and 55% in alc. content. The sap of the nipa palm is the cheapest source of alc. in the world, namely, \$0.027 per l. of 180 proof, or 90% alc. as compared with \$0.058 dollars for sugar beets, and 0.037 for potatoes. With additional economies 93% high grade alc. could be produced at \$0.035-0.0375 per l. The nipa palm gives great promise of being a source of refined sugar (sucrose). A description of the nipa palm and particulars about the uses of the sap are given. The sap has approx. the following comp. stated in g. per 100 cc.: D<sup>150</sup> = 1.0720; total solids = 18.00; ash = 0.48; acidity = trace; sucrose - 17.00; reducing sugars - trace. C. A. NOWAK.

Preliminary Reports on the Kropff Malting System. C. Bleisch. Allgem. Z. Bierbr. Malsfabr., 39, 281-4, 291-4, 299-301.—This system is based upon the pneumatic principle thruout, the malt being placed in boxes, which are closed at intervals for some time so as to exclude air and permit a considerable accumulation of  $CO_3$ . The author's experience in practice with this system has so far been fairly satisfactory, altho a certain amt. of skill in working is necessary. The loss in malting is reduced by about 2 I/2% calc. on dry substance, floor space necessary is minimized and cost of labor reduced. The author recommends putting this new system extensively to practical test with a view to detg. whether the maltsters are confronting a fundamental innovation in malting which will contribute to perfect their product and reduce expenses.

C. A. Nowak.

The Working up of the Malts of Last Season's Harvest with Special Attention to the Crushing. F. SOEDING. Z. ges. Brauw. 34, 357-9.—The author discusses the difficulties encountered in working with the year's malts owing to the higher albumen content and the finer crushing. Suggestions are made for conducting the mashing process in a manner which will overcome these difficulties. C. A. NOWAK.

The Determination of Starch in Spent Grains. C. KREUZER. Z. ges. Brauw., 34, 277-84, 290-3.—1. None of the customary mashing methods employed in practice or in the lab. permit a complete utilization of the starch. 2. The detn. of starch throws a light upon the influence of the various brewing operations upon its utilization. 3. The analysis of lab. grains yields a comparatively high % of starch. 4. The method of Seibriger for detg. the barley ext. does not open up all the available starch altho the % remaining is very small. The author has worked out a method for detg. the

starch in the grains using  $H_2SO_4$  which requires only a short time for its execution. Owing to the small quantity of material under exam. it is necessary to make 2-3 detns. in order to arrive at the mean value.

C. A. Nowak.

The Saponins (KOBERT). 17.

Determination of Glycerol (Ross). 12.

Action of Ultraviolet Rays on Saponin (SOLACOLU). 11.

Ethereal Oils (Schimmel & Co.) (HAENSEL). 17.

Ger., 235,660, Oct. 1, 1908. V. SLAVICEK, Wien. In the continuous distillation and rectification of alcoholic liquids, the crude vapors are sepd. and the phlegms from the 1 part, purified by the first running, are led, uncooled, before the beginning of the rectification, directly and in the same direction into the part of the downwardly directed crude vapors, contg. the last runnings, and which have likewise been purified by the first runnings.

Ger., 235,877, June 24, 1909. H. OETTINGER, Friedenau-Berlin, and F. HIRSCH, Dortmund. Decreasing the nitrogen content and increasing the extraction yield of barley brew during the softening process, by transmitting an elec. current of large amperage thru the fluids in which the material is suspended.

# 17. PHARMACEUTICAL CHEMISTRY.

#### VIRGIL COBLENTZ.

Examination of a Crystallin Deposit from a Fluid Extract of Cinchona. The Determination of Cinchona Alkaloids and Quinine Silicotungstate. Javillier and B. Gurrithault. Bull. sci. pharmacolog, 18, 93-6.—A cryst. deposit found in an extract of cinchona (18 months old) made by percolation with glycerol, EtOH and H<sub>2</sub>O according to the U. S. P. process consisted entirely of Ca quinate. Alkaloids were absent. The alkaloids in the soln. were detd. by the Yvon silicotungstate method with satisfactory results. The comp. of the quinine silicotungstate was found to be, at 30°, SiO<sub>2-12</sub>WO<sub>3-2</sub>H<sub>2</sub>O<sub>2</sub>C<sub>20</sub>H<sub>20</sub>O<sub>2</sub>N<sub>3</sub> + 7H<sub>2</sub>O. Six H<sub>3</sub>O mols. are lost at 120°. It is a pale, amorphous, yellow powder of constant comp.

Assay of Pyramidon. G. PATEIN. J. pharm. chim., 3, 166-7.—The author calls attention to the fact that the 1908 Codex in its assay method neglected to require the addition of H<sub>2</sub>O before making alk. with NH<sub>4</sub>OH as recommended by him (J. pharm. chim., 22, 5, (1905)). If this be neglected the formation of the double hydrochloride of NH<sub>4</sub> and pyramidon which is apt to cryst. out would tend to indicate the presence of antipyrine.

V. K. Chesnut.

Variation in the Composition of Gentian Root during a Year's Growth. Marc Bridgi. J. pharm. chim., 3, 294-305.—The comp. of the root was found at different seasons to suffer considerable change especially with regard to the carbohydrates hydrolyzable by invertin (1.2 in spring to 7.8% when mature). The gentiopicrin, which was sepd. from the carbohydrates by means of AcOR, suffered little change, generally amtg. to 2%. Gentianose, generally present to the extent of 3-5%, suffered some diminution in May and June when it was partially replaced by gentiobio e and increased to the greatest content in Aug. and Sept. Sucrose suffered the greatest variation. It accumulated in the root in winter but disappeared in greater part together with the gentianose when growth began in the Spring.

V. K. Chesnut.

Assay of Pepsin. L. PORTES. J. pharm. chim., 3, 341-3.—In the assay process for pepsin given in the 1908 Codex, the directions to allow the products of digestion

to cool to ordinary temp. before testing with HNO<sub>2</sub> are too vague and lead to contradictory results as shown by expts. carried out on the same digestion mixture tested at once and after cooling to 15° and 10°. The author much prefers to make the digestion on a water bath than in an air bath because the temp. can thus better be regulated.

V. K. Chesnut.

Action of Hydrogen Peroxide upon Digitalis Powder. E. CHOAY. J. pharm. chim., 3, 343-45.—Digitalis leaves dried in vacuo, at room temp. in the open air, and at 40° and afterward powdered were tested with H<sub>2</sub>O<sub>2</sub>. The prep. dried in vacuo was found to be 20 times as active in decomposing H<sub>2</sub>O<sub>3</sub> than that dried at 40°. The air dried prep. had an intermediate activity.

V. K. Chesnut.

Remarks on the Leaves of Uva Urai and of Box. M. Thouvenin. J. pharm. chim., 3, 436-40.—Contrary to the view expressed in the 1908 Codex, the leaf of the box, Buxus sempervirens can not be distinguished in powder form from that of uva ursi by the absence in the former of simple prismatic oxalate crystals for these are present in the leaf of the latter a little distance from the nerves. The leaves are most easily distinguished by the stomata on the under side of the leaf. Those of the box are 0.028 by 0.025 mm. while those of uva ursi are 0.04 by 0.043. If total substitution by box be suspected, test the infusion with FeSO4 and AcONa. Uva ursi leaves with FeSO4 give a blue ppt. and with AcONa a violet ppt. These tests are negative with infusion of box leaves.

Observations on the Desiccation of Cis-Terpene Hydrate.  $(C_{10}H_{20}O_2 - H_2O)$ . LEULIER. J. pharm. chim., 3, 440-1.—Cis-Terpene hydrate is slowly but completely volatil at 100°. It can be dried without loss of wt. in vacuo over  $H_2SO_4$ . V. K. C.

Preservation of Juices, Extracts and Similar Preparations. Lefeld. Berlin. Pharm. Ztg., 56, 333; thru Chem. Zentr., 1911, I, 1647.—The author proposes to preserve such preps. by keeping them in an atm. of alc. and has constructed 2 special, bottle stoppers for this purpose. These are described with drawings. E. J. Crane.

Tryparosan, Trypan Red, Trypan Blue and Parafuchsin in Immunization against Rabies. CLAUDIO FERMI. Ist. Antirab., Univ. Sassari. Arch. jarm. sper., 11, 256-9.

—The results were for the most part negative.

A. W. Dox.

A New Adulteration of Resin Scammony. P. Guiguss. Ann. fals., 4, 397.— The product was clear brown, slightly opaque, sol. in Et<sub>2</sub>O, insol. in water, hot or cold and left only a powdery residue insol. in alc. Analysis showed H<sub>2</sub>O, 4.50%; insol. in Et<sub>2</sub>O, 11.48%; sol. by diff., 84.02%. Rotation of the pure, decolorized resin was 22° 58'. Wt. of resin in 100 cc. 4.64 g. Observed deviation 28°. The residue insol. in Et<sub>2</sub>O and alc. was easily identified and consisted of the finely powdered root of scammony. One may identify the starch grains of 10 to 12  $\mu$  in groups of 2 or 3. The cellular particles also serve, as do the rhombohedral crystals of Ca oxalate. The conclusion reached was that the product consisted of industrial resin with powdered scammony root.

Ethereal Oils. Schimmel & Co. Milititz, near Leipzig. Trade Circular, April, 1911; thru Chem. Zentr., 1911, I, 1837-9.—The yield of oil from Alpinia galanga is 0.04% of the fresh rhizomes. The oil is light yellow and it has a pleasing odor;  $d_{20}$  0.968;  $\alpha_B^{27.5}$  about 6°. It contains Me cinnamate (48%), cineole (20-30%) and probably d-pinene and camphor. The leaves of this plant also furnishes small quantities of an ethereal oil which probably contains Me cinnamate. Andiopogon (Cymbopogan) schoenanthus var. nervatus, from the Sudan Yield, 1.9% of a brown oil having a feeble odor;  $d_{10}$  0.9405;  $\alpha_D$  26° 22';  $\alpha_D^{20}$  1.49469; "acid number," 4.6; "ester number," 9.3; "acetyl ester number," 99.1. It dissolves in 0.5 vol. and more of alc. (80%) and in dil. soln. deposits paraffin. Oil, from Angelica (Militiz) had  $d_{10}$  0.8550-0.8767;

 $\alpha_{\rm D}$  28° 2'-20° 11';  $n_{\rm D}^{90}$  1.47782-1.48316; acid number, 0.5-1.6; ester number, 17.6-21. It gives an opalescent soln. in 6-2.5 vols of alc. (90%). Oil from Cinnamomum parthoxylon Meissn. The wood (15.82 kg.) of this tree, from Java, gave 124 cc. of pale yellow oil, consisting chiefly of safrole; d<sub>10</sub> 1.0799; α<sub>D</sub> 1° 22'; π<sup>20</sup> 1.53229. It dissolves n 2.6 and more vols. of alc. (90%). The bark of this tree has been stated to be free from ethereal oil. Oil from Citrus hystrix D C (C. papedia Miq.). The fruit rinds, from Java, gave 4% of oil; dm 0.942; ap 9°. It contains 40% of citral. Curcuma oil. The yield of golden-yellow oil from turmeric roots was 3.23%; d1, 0.9430; ap 23°;  $n_{\rm p}^{20}$  1.51236; "acid number", 1.6; "ester number", 7.8; "acetyl ester number," 30. It exhibits the phellandrene reaction, is insol. in 10 vols. of alc. (80%), but dissolves in 0.4 vols of 90% alc. Orange oil. The following constants have been determined from pure specimens. Bitter oil,  $d_{13}$  0.8527 (usually 0.854-6);  $\alpha_{D}^{20}$  89° 30′ to 94° (the first 10%) of the distillate shows a higher refraction power than does the original oil);  $n_n^{20}$  1.473-5; residue after evaporation, 3-5%. Sweet oil,  $d_{18}$  0.848-53;  $\alpha_D^{20}$  95° 30′ to 98° (the first 10% of the distillate has a refractive power but little, if at all, lower than that of the original oil); n<sup>20</sup> 1.473-5; residue after evaporation, 1.5-4%. Oil from Savin wood contains  $\alpha$ -terpinene and sabinene; b. 175-7°;  $d_{10}$  0.8469;  $\alpha_D$  0° 37′;  $n_D^{20}$  1.47137. It failed to form a tetrabromide, but with NO gave  $\alpha$ -terpinene nitrosite (m. 156°). Sandal wood oil from India, contains, in the fraction b. 210-20°, teresantalol (m. 112-4°). Spanish hops, Majorana onites L. Benth. (Origanum onites L., O. smyrnalum L.), from Smyrna, gave 2\% of a dark brown oil; d<sub>14</sub> 0.9572; \alpha\_D -1° 30' or -2° 10'. It contained 70% of phenols, and is sol. in 2.2 vols. or more of 70% alc. It differs from the ordinary com. Smyrna oil in its much higher phenol and lower linalool content. The phenols consisted almost exclusively of carvacrol; after their removal the residue was found to contain α-pinene, p-cymene, l-linalool and d-camphor. Indian turpentine oil, from Pinus longifolia, differs in composition from ordinary terpentine oil. It is particularly characterized by its high silvestrene content (hydrochloride m. 71-2°); it contains a little α-pinene and, in the high b. fraction, there is present a sesquiterpene; d<sub>10</sub> 0.9371;  $\alpha_D$  37° 4';  $n_D^{20}$  1.50252. Hydrochloride, large needles, m. 59.5-60.5°. Balan oil, from the Java plant Heritiera littoralis Ait. (Balang passisir). Yield, 0.05%. Brown oil with an intense odor resembling that of orange oil; dis 0.9042; no 1.47715; "acid number", 13.0; "ester number", 20.5. It is sol. in 0.5 vol. and more of 90% alc. and it gives, with NaHSO<sub>2</sub>, an additive compound, which has an odor of decylic aldehyde and is probably not homogeneous. The leaves of Eugenia occlusa Kurz (Syzygium occlusum Miq.), from Java, gave 0.05% of a dark brown oil; d<sub>18</sub> 0.9567;  $\alpha_{\rm D}$  —1° 40′;  $n_{\rm D}^{20}$  1.48614. It dissolves in 0.5 vol. and more of 90% alc. and at about 12°, the soln. deposits crystals, possibly of paraffin. The oil contains fairly large quantities of citral and of other aldehydes. Oil from the fruit of Pelea madagascarica Baill. (?). Yield, 4.05%. The oil was yellow and had an odor of anisic aldehyde;  $d_{18}$  0.9553;  $\alpha_D$  32° 22';  $n_D^{20}$  1.51469. It dissolves in about 4 vols. and more of 80% alc. and contains a little anethole, but it consists chiefly of anisic aldehyde, together probably, with other aldehydes. The cones of the blue pine, (Pinus excelsa Wall.), from India, gave a pale yellow oil;  $d_{10}$  0.8757;  $\alpha_D = 32^{\circ}$  45';  $n_D^{20}$  1.47352; acid number, 0.5; ester number, 5.6, corresponding with 2% of bornyl acetate. The oil is soluble in 5 vols. or more of 90% alc. In the case of certain specimens of bergamot oil, the difference between the "hydrolysis number" and the "acid number II" has been found to be as great as 7, but it has never exceeded 10, which value indicates adulteration. The difference between the hydrolysis number and the acid number II of lavender and of "petit grain" oils is o-3. The investigation of glyceryl esters has been continued by a somewhat different method. The oil under examination (10 cc.) is shaken out with 5% alc. (20 cc.) (no petroleum ether being used); the layers of liquid are allowed to become as clear as possible and the filtrate (10 cc.) is neutralized, in presence

of phenolphthalein and then hydrolyzed during 1 hr. by means of 0.5 N KOH (5 cc.). In the case of the pure oils the quantity of alkali which combines does not exceed 0.1 cc. of a 0.5 N KOH soln.

J. BISHOP TINGLE.

Ethereal Oils. HEINRICH HAENSEL. Pirna. Trade Circular, Oct., 1910, April, 1911; thru Chem. Zentr., 1911, I, 1839; cf. C. A., 5, 1156.—Lovage oil: Yield, of oil from the roots, 0.7%; d. 1.053. Spanish myrtle oil:  $d_{18}$  0.9406;  $\alpha_D$  23.04°; hydrolysis number, 128; after acetylation, 159. The oil, after being freed from terpenes, had  $d_{15}$  0.9713;  $\alpha_D$  25.48°; hydrolysis number 195; after acetylation, 234. This substance is a much superior preparation. Peppermint oil from dried plants from the Palatinate; yield, 0.36%. It is a light brown oil with a penetrating, delicate odor and it does not give a deposit at -14°; d<sub>22</sub> 0.9076; acid number, 3.73; ester number, 33.6 = 9.36% of esterified menthol; ester number after acetylation, 147.46 = 46.15% of total menthol. Russian peppermint oil: Light yellow oil with an agreeable, delicate odor; it gives no deposit at -14°; d<sub>20</sub> 0.9031; acid number, 1.87; ester number, 22.4 = 6.24% of esterified menthol; ester number after acetylation, 141.2 = 43.98% of esterified menthol. Oil from the rind of Rhus aromatica, obtained by steam distillation under reduced pressure; yield, 0.07%. Dark brown oil with a sharp, aromatic odor, wh.ch, after a time, changes to that of tar; when cooled the liquid becomes viscid and freezes -15°; d. 0.954. The oil has an acid reaction; it contains a phenol, palmitic acid and probably decatoic acid, but no constituent which could be sepd. by adding "bisulfite." The yield of oil from the blossoms of Lamium album, extracted by means of steam, under reduced pressure, was 0.537%. It is a brown oil with a pleasant odor and it deposits crystals at the ordinary temp. J. BISHOP TINGLE.

The New Remedies and Pharmaceutical Specialties of 1910. F. Zernik. Suddeut. Apoth. Ztg., 51, 26-7, 34-6, 42-4.—A review.

A. Vorisek.

The Saponins. KOBERT. Suddeut. Apoth. Ztg., 51, 158-9.—With the exception of solanine which is the connecting link between the alkaloids and saponins, the latter are N-free glucosides. Most of them are neutral substances; some are acid in reaction. Pb subacetate ppts. all saponins, Ba(OH), reacts with most of them, but only the acid ones are pptd. by Pb(OAc)<sub>2</sub>. They also combine with cholesterol and phytosterol. Saponins are amorphous colloids, they dialyze with difficulty and several of them may be salted out of soln. Acids and alc. interfere with their foam-forming property. For purification, hot alc. is used after tannins and colors have been removed with Mg(OH)<sub>2</sub>. Most saponins are insol. in cold abs. alc. but such dissolve in warm alc. containing H<sub>2</sub>O. They are grouped into those (1) easily sol. in cold H<sub>2</sub>O; (2) sol. in H<sub>2</sub>O made alkaline; (3) insol. in pure form but sol. in the presence of water-sol. or impure saponins. When saponified most of them yield glucose and sapogenin which when sapon. gives glucose and "endosapogenin." Not all saponins are poisonous since drugs containing them, like sarsaparilla are extensively used. All known saponins should fall into 5 general groups having the formulas:  $C_nH_{2m-8}O_{10}$ ;  $C_nH_{2m-16}O_{28}$ ;  $C_nH_{2m-6}O_7$  (secondary glucosides from saponins);  $C_nH_{2n-6}O_2$ ; (endosaponins) and  $C_nH_{2n-6}O_2$ ; (oxysapogenols). Many saponins react with AuCl<sub>2</sub>, NH<sub>2</sub>-AgNO<sub>2</sub>, K<sub>2</sub>FeCN<sub>6</sub>, FeCl<sub>2</sub>, HgCl<sub>2</sub>, reduction taking place on warming. They decolorize KMnO4 soln. and when warmed with Nessler's reagent cause production of yellow color and turbidity, the mixture often gelatinizing. When pure they do not react with Millon's reagent. With H<sub>2</sub>SO<sub>4</sub> a red to violet color is developed on warming; the soln. in some cases becomes fluorescent. Many react with Cu in alkaline soln., a green, gelatinous compd. sol. in H<sub>2</sub>O separating.

The Significance to Medicin and Arts of Compounds Containing Available Oxygen. RUETHE. Suddeut. Apoth. Ztg., 51, 214.—The uses of H<sub>2</sub>O<sub>2</sub>, Na<sub>2</sub>O<sub>3</sub>, NaBO<sub>2</sub> and ZnO<sub>3</sub> are enumerated.

A. VORISEK.

Determination of the Specific Gravity of Wax. G. Fromms. Apoth. Ztg., 26, 402.—The prep. of small wax balls for the indirect detn. is difficult if the directions of the German Ph. Vol. V (dropping melted wax from a glass rod) are followed. It is better to prepare a capillary tube 1.2-2 mm. from a 6-8 mm. tubing. A rod of wax is then introduced into the tube and on heating, the melted wax allowed to drop into a test tube containing  $\frac{3}{2}$  of alc., the upper portion of which is kept warm by heating over a small flame. Under these conditions the particles of wax remain round when they sink into the colder alc. layer and solidify.

A. VORISEK.

Essential Oils. I. Essence of Orange Blossoms. G. LALOUR. Bull. soc. chim., 7, 1101-7.—When orange blossoms are steam-distilled, certain constituents are modified or destroyed the terpene esters undergoing sapon. Such changes affect the optical properties of the product. In comparing the oil from spring and fall flowers the flowers were exhausted with petroleum ether (3 washings). After removal of the solvent, of which last traces were removed in vacuo, the residue was steam-distilled. The oil layer in the distillate was decanted off, and the aq. layer, after supersatn. with sea salt, washed with Et<sub>2</sub>O. The 2 products kept separate. The nonvolatil residue, mainly wax, was deprived of H<sub>2</sub>O by expression, repeated trituration with alc. and drying in vacuo; m. p. 45-60°, sapon. no. 80.1 (summer oil), 77.2 (fall oil). It dissolved in petroleum ether, CaHa, CHCla, and incompletely in 90% alc. Of the spring flowers, 1000 parts yielded 2.272; of the fall flowers, 1.795 pts. of the wax and 1.4726 and 1.3317 parts of oil, resp. The oil obtained by extraction or distillation under reduced pressure was l-rotary, that distilled under ordinary pressure being d rotary. The av. for the latter is  $+5^{\circ}$ . The oil from fall flowers had a higher value than the spring oil  $(+9.8^{\circ})$ . The May oil is richer in Me anthrapilate, the value found, 3.53% (per mill?) being one half of the av. This low value is attributed to the severe preceding winter. To determin the esters and terpene alcs., sapon. should be carried out after the removal of Me anthranilate. For this the oil is dild. with 6 vols. of Et<sub>2</sub>O, cooled to -4° and treated with H<sub>2</sub>SO<sub>4</sub> dissolved in Et<sub>2</sub>O, filtered and kept at o°. The ethereal layer is decanted, washed with H<sub>2</sub>O and dehydrated. Results of the detns. are tabulated. The nerol and geraniol contents were obtained by esterification with phthalic anhydride in the presence of benzine. The May oil is poorer in esters and in total alc., the free alcs. of the oils not differing. In comp. the oil from spring flowers bears similarity with the oil from young branches, while the oil from fall flowers is like that from the older branches. The differences between the 2 oils confirm the theory that terpenes are formed in the green parts of plants. Me anthranilate, which is found only in the flowers, is thought to be a product of decomposition of a glucoside.

A. Vorisek.

Study of the Essential Oil of Mespilodaphne pretiosa. ROURE-BERTRAND FILS. Bull., [3] 2, 19–23.—Only the female plants, the so called "female priprioca" yield the essential oil. Oil of the Branches: The oil, mostly linaloöl, of which the yield by steam distillation is about 0.5%, is a very mobil yellow liquid of an odor resembling that of linaloes. It is sol. in all proportions in 80% alc. and in 1.25 or more vols. of 70% alc.;  $d_{10}$  0.8912,  $[\alpha]^{100}$  +7° 20′,  $n_D^{20}$  1.469, acid no., 1.4; sapon. no., 13.3; esters, as linalyl acetate, 4.65%; sapon. no. of the acetylated oil, 165,2; alcohols, as linaloöl, 51.8%. Oil of the Wood: The wood yields about 0.7% of an oil which sepd. into 2 layers. The portion lighter than  $H_2O$  is greenish yellow, has odor of linaloe with traces of cinnamic alc. Sol. in 0.5 vol. 80%, and in 1.5 vol. 70% alc., the soln. remains clear on addition of more of the solvent;  $d_{10}$  0.9539,  $[\alpha]^{100}$  +8° 48′,  $n_D^{20}$  1.501, acid no., 0.7; sapon. no., 100.8; esters, as linalyl acetate, 35.28%; sapon. no. of the acetylated oil, 205.1; alcohols as linaloöl, 66.6%. The heavier oil, mostly acetate and benzoate esters, is pale brown, its odor recalls cinnamon and cinnamic alc. Sol. in 4 vols. of 80%,

and insol. in 70% alc. Readily sol. in alc. KOH with a fine golden yellow color. Remains liquid at  $-15^{\circ}$ ,  $d_{10}$  1.0551,  $[\alpha] + 3^{\circ}$  8′,  $n_D^{20}$  1.545, acid no., 3.5; sapon. no. of the acetylated oil, 247.8.

A. VORISEK.

An Alkaloid of Eschscholtzia californica. G. Brindejonc. Bull. soc. chim., 9, 97–100; thru J. Soc. Chem Ind., 30, 152.—About 0.25% of an alkaloid,  $C_{10}H_{20}N_4O_4$ , termed ionidine, was obtained from the root of the plant. From alc. it cryst. in flattened colorless prisms, m. 154–6°, permanent in the air. In cold 90% alc. it dissolved to 0.46%, in boiling alc. to 7.0%; 1 pt. is sol. in 2500 of water. Picric acid, AuCl<sub>3</sub>, and PtCl<sub>4</sub> ppt. it from soln. but no cryst. salts were obtained. With H<sub>2</sub>SO<sub>4</sub> and a trace of a nitrite a violet color is produced, similar reaction being obtained with Froehde's reagent.

A. VORISEK.

Contribution to the Chemistry of Anesthetics. III. Nitrous Oxide. C. BASKER-VILLE AND R. STEVENSON. J. Ind. Eng. Chem., 3, 579-82.—The first part of the work treats of the prep. analysis and purification of N<sub>2</sub>O, and contains a complete list of literature references. The methods of analysis of com. N<sub>2</sub>O are fully described. In the method devized by the authors, the N<sub>2</sub>O is caused to react with reduced Cu in an atm. of H, the CuO then reduced by H, and the H<sub>2</sub>O formed collected in a CaCl<sub>2</sub> tube for weighing. Results obtained show that compressed nitrous oxide, supplied by American manufacturers, consists of 95.9-99.7% of N<sub>2</sub>O, 0.16-3.94% of N<sub>2</sub>O. 0.13-0.16% of H<sub>2</sub>O, none or traces of NH<sub>2</sub>, some O<sub>2</sub> and no CO<sub>2</sub>. Nitrous oxide to be used for anesthesia should contain at least 95% N<sub>2</sub>O, and be free from solids, liquids, combustible organic matter, Cl, and other oxides of N<sub>2</sub>.

A. VORISEK.

The Quantitative Determination of Ketones in Essential Oils. E. K. Nelson. J. Ind. Eng. Chem., 3, 588-9.—Pure ketones were used in expts. carried out to test the accuracy of the Walther's method. In this a ketone is boiled under a return condenser with a standard alc. soln. of NH<sub>2</sub>OH in the presence of NaHCO<sub>2</sub>. To the cooled mixture HCl is added, then water to definit vol. In an aliquot of the filtrate the acid is neutralized with 0.5 N NaOH using Me-orange. The unconsumed NH<sub>2</sub>OH is then titrated with 0.1 N NaOH and phenolph. Results are fairly satisfactory except with fenchone. To use the method for detn. of a ketone in an oil, the influence of the other constituents of the oil must be considered. At present the method is available for oils containing carvone, camphor, pulegone or thujone.

Fatty Acids of Codliver Oil. A. Heiduschka and E. Rheinberger. Lab. d. K., Univ. München. Pharm. Zentralhalle, 52, 837-8.—Dehalogenation of the tetrachlorotetraiodoterapinic acid, C<sub>17</sub>H<sub>22</sub>O<sub>2</sub>(CII), prepared from the cod-liver oil fatty acids, and reported previously (Ibid., 51, 203) was accomplished by using Zn+AcOH; the product was purified by distillation (154°). Its I no. 264.5, does not correspond to terapinic acid (387.4) though it comes close to the hydrated acid, C<sub>17</sub>H<sub>22</sub>O<sub>2</sub>, (288.3). The tetrachlorotetraiodo compd. is changed into chlorotromo derivative by heating with an excess of Br in a sealed tube at 100° for 24 hrs. It dissolved in volatil solvents, and cryst. from EtOAc. m. 150°. The Octachloroterapinic acid gave a potassium salt when heated with alc. KOH. Linoleic acid forms trichlorotriodo derivative, C<sub>16</sub>-H<sub>20</sub>(CII), white and cryst., m. 95°.

Genesis of Alkaloids in Plants (CIAMICIAN, RAVENNA). 11.

Essence of Bupleurum fructicosum Lime (FRANCESCONI, SANNA). 11.

Codliver Oil (LOUISE). 27.

Determination of Malic Acid (DUNBAR, BACON). 12.

Lactose (BURR, BERBERICH). 12.

Diabetic Foods (ARNAUD). 12.

Toxicity of Strophanthin (DANIELOPOLU). 11.
Strychnine Alkaloids (LEUCHS, ANDERSON). 10.
Morphine (WIELAND, KAPPELMEIER). 10.
Antirol (THOMS, SIEBELING) 10.
Dihydroanethole (THOMS, DRAUZBURG) 10.

Ger., 235,625, Feb. 13, 1909. KALLE & Co., Biebrich a/Rh. Colorless compounds of 3-hydroxy-(1)-thionaphthenes with aldehydes, ketones, and diketones are obtained by heating 3-hydroxy-(1)-thionaphthene or its derivs. substituted in the benzene nucleus or its homologs with aldehydes, ketones, or diketones in the presence of suitable solvents which do not impart color to these compds., such as alc. Examples are given.

Ger., 235,801, Nov. 5, 1903. E. Merck, Darmstadt. In the manuf. of CC-dialkylthiobarbituric acids, employing for the condensation of dialkylmalonic esters with thiourea, the free alkali metals or their amides instead of the metal alcoholates.

Ger., 235,802, Dec. 1, 1964. Idsm. Mfg. CC-dialkyliminobarbituric acids (5-dialkyl-2-imino-4,6-dihydroxypyrimidines) by heating dialkylmalonic esters with guanidine without the addition of a condensing agent. Examples are given.

Ger., 235,836, Apr. 26, 1910. CASSELLA & Co., Frankfurt a/M. Production of halogen substitution products of the indophenols from carbazoles and p-nitrosophenols and their leuco derivs. In order to introduce halogen into the condensation products, which are derived from carbazole or N-alkylcarbazole and p-nitrosophenols, either suitable halogen derivs. of carbazole can be condensed with p-nitrosophenol or halogencontg. p-nitrosophenols can be combined with carbazole or halogen carbazoles. The p-chloro- and p-bromophenols can be readily produced technically by the nitration of p-chloro- or p-bromophenol.

Ger., 235,955, Mar. 20, 1910. C. N. OTIN and B. WASER, Charlottenburg. In the manuf. of organic compounds by electrolytic reduction, the cathodes are rendered especially active by giving them a porous structure and filling the cathode chamber with them as completely as possible. The entire cathode chamber is filled, not with metal powder, but with the metallic cathode material in the form of granules, wires, or the like. Further details are specified and examples of application are given.

Ger., 236,045, Dec. 4, 1909. Addition to 223,305, May 22, 1908; cf. C. A., 4, 2980. C. F. BOEHRINGER & SÖHNE, Mannheim-Waldhof. Manuf. of esters of digly-colic acid with phenols and phenol derivatives. The esters produced by the principal process can be obtained more simply by acting on the diglycolic acid or its salts, and phenol, with acid condensing agents such as POCl<sub>2</sub>, PCl<sub>3</sub>, or COCl<sub>2</sub>. The process is applicable in the heat or in the cold, in the latter case adding a tertiary organic base such as dimethylaniline or pyridine, and also an indifferent solvent.

Ger., 236,046, July 15, 1910. ANILINFARBEN- & EXTRAKT-FABRIKEN VORM. JOH. RUD. GEIGY, Basel. Manuf. of unsymmetrically substituted diphenylmethans derivatives. If chloromethyl alc., chloromethyl ether, or similarly acting agents are allowed to act on o-cresotinic acid in the presence of high % fuming HCl, the group CH<sub>2</sub>.Cl enters in the p-position to the OH group, yielding the p-chloromethyl-o-cresotinic acid (I). Upon warming with H<sub>2</sub>O all the Cl is split off as HCl, whereby a portion of the p-chloromethyl-o-cresotinic acid goes over into the p-hydroxymethyl-o-cresotinic acid (II) crystallizing from the filtrate. The other portion of (I) is converted into an anhydro compd. of (II) and remains on the filtrate. This conversion takes place to a small extent in the cold by washing out the crude product filtered from

the strong HCl soln. (I) with the splitting off of HCl and evolution of considerable heat, combines with aromatic mono- and dialkylamines or their nucleus substitution products with free p-position, whereby unsymmetrically substituted diphenylmethane derivs, result which react basic as well as acid.

# 18. ACIDS, ALKALIES, SALTS AND SUNDRIES.

### T. LYNTON BRIGGS.

Chemical Research and Industrial Progress. W. R. WHITNEY. Trans. Am. Electrochem. Soc., 19, 17-30.—The moral obligation on the part of large industrial organizations having fully equipped research laboratories to publish results obtained therein is strongly emphasized.

C. N. MOORE.

An Acid Tester. PH. EYER. Glashatte, 41, 603.—A patented cylinder for measuring the strength of pickling acids. Ten cc. of the acid are measured into the cylinder and roughly titrated with N NaOH. When the ppt. of iron in the acid is permanent, the strength of the acid is obtained by reading the height of the liquid upon the scale of the cylinder.

J. B. PATCH.

Manufacture of Sulfuric Acid by means of Towers. Anon. Ind. chim., 11, 137-9.

—Brief description of the system of H<sub>2</sub>SO<sub>4</sub> manuf. in which towers are substituted for the usual Pb chambers. In such a system, to convert all the SO<sub>2</sub> from 10 tons pyrites a day into H<sub>2</sub>SO<sub>4</sub>, all the HNO<sub>3</sub> being recovered with d. 30-5° Bé., a total of 200 cu. m. space is required, only about 30 cu.m. being used for the conversion of SO<sub>2</sub> into H<sub>2</sub>SO<sub>4</sub>. This can be reduced to 120 cu. m. if, instead of absorbing in the last tower the traces of N oxides, with H<sub>2</sub>O, H<sub>2</sub>SO<sub>4</sub> of 59° Bé. is used, 5-10% of the HNO<sub>3</sub> being lost in that case. Acid of 56-8° Bé. can be obtained by this system.

Chas. A. ROUILLER.

Direct Absorption of Ammonia. M. GREBEL. J. Gas Lighting, 114, 1006-7; Rev. gén. chim., 14, 233-9; cf. C. A., 5, 3145.—The crude hot gases, used for reheating the sulfate saturators, are relieved of their tar content, which is in the form of a mist, by injecting hot tar into them, the condensed mist and tar being finally collected in a receiver. The hot tar-free gases are next led into the sulfate saturator by an injector, which dips slightly into the acid and which prevents the exit gases from carrying away acid. The mother liquor is circulated thru the saturator until satd., when it is run into separators and relieved of small amts. of floating tar. From here the cryst. salt is centrifugalized and passed on to a dryer.

C. A. C.

New Method of Extraction of Sulfur from Minerals Containing it in the Native State. Anyonio de Gregorio Rocasolano. Saragossa. Rev. real acad. cien. Madrid, 8, 895-912; Anales soc. espan. fis. quim., 8, 264-79.—Discussion and criticism from an economic standpoint of the calcaroni, the processes of Gill, Thomas, Fresch, Dubreuill, Condy Bollman, and the Neopolitan process for the extraction of S. The author first attempted to modify Thomas' process, using air (heated above the m. p. of S by means of a tubular system thru which circulated steam at 7 atms. pressure) as a heat vehicle instead of superheated steam. This modification was open to most of the objections to the Thomas process and, morever, cannot be recommended because of the small heat capacity of air compared to the heat capacity and heat of

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fusion of S. The following process was then devized and is now in use industrially in several parts of Spain. This method secures extraction of S and refining of part of it with a single fusion. A cylindrical heater (holding about 640 kgs. of mineral and having an opening near the top for loading) is inclined at an angle of 40-45°. The mineral is held in place by a movable grating near the lower end and below this grating the cylinder is terminated by an elbow dipping under water. When heat is applied a portion of the S melts and, carrying with it organic bituminous material volatil at 120-130°, flows out the elbow of the cylinder and is collected under water as fused black S of 97-98.5% purity. As the temp. rizes, S is volatilized and carried thru the upper end of the heater into a cool chamber (where it is obtained as flowers of S) and also thru the lower end of the cylinder (where it is collected in yellow masses of 99.2-99.7% purity). Air is excluded from the app. by means of water seals at all openings. All the principal com. forms of S may thus be obtained in 1 operation and the proportion of each form may be varied at will within certain limits. The following is a typical single operation: from 600 kgs. of mineral containing 30% S were obtained 12 kgs. flowers of S and 155 kgs. of fused S of the 2nd and 3rd commercial grades, making a total yield of 92.7%. H. S. PAINE.

Atmospheric Saltpeter. Anon. Sudduet. A poth. Ztg., 51, 321.—A discussion of the production of nitrates from atmospheric  $N_2$ .

A. VORISEK.

The Ammonium Nitrate Plant in Notodden. F. A. Buhler. Gross-Lichterfelde. Chem. Ind., 34, 210-2.—A detailed description of the manuf. of NH<sub>4</sub>NO<sub>2</sub> to be used for safety explosives. A complete set of drawings of the plant is given.

E. J. CRANE.

By-products of the Industries. NARINOLI. Ind. chim., 11, 124.—Criticism of the paper by Fachini (C. A., 5, 2309).

CHAS. A. ROUILLER.

By-products of Certain Industries. S. Fachini. Milan. Ind. chim., 11, 172-3.

—Answer to Narinoli (cf. preceding abstr.). Chas. A. Rouiller.

Activity of the Berufsgenossenschaft der Chemische Industrie in the Field of Accident Prevention in 1910. Chem. Ind., 34 (supplement to No. 14, 1-50).

E. J. CRANE.

Ger., 235,751, Apr. 24, 1909. A. WERNICKE, Halle a/S. In the production of potassium chloride, the cryst. mass obtained in the continuous crystn. process is directly and uninterruptedly led counter to the mother liquor in order that the lowering of the temp. due to the surface liquor on the crystals may effect a further sepn. of the KCl.

Ger., 235,765, Dec. 29, 1909. Addition to 235,300, Sept. 16, 1909; cf. C. A., 5, 3137. Bad. Anilin- U Soda-Fabr., Ludwigshafen a/Rh. In the manuf. of ammonia, employing Al-N compds. which contain any admixts., instead of N compds. of other elements. In this manner, besides a ready production of NH<sub>2</sub>, a very pure Al<sub>2</sub>O<sub>2</sub>, suitable for the manuf. of Al, can be obtained from impure Al-N compds. such as are obtained technically from cheap impure alumina, as crude bauxite. Modifications are specified.

Ger., 235,766, Jan. 25, 1910. Addition to 235,300, Sept. 16, 1909; previous addition 235,765; cf. supra. Idem. In the manuf. of ammonia, such quantities of bases, or such kinds of bases, are added to the mixt. contg. the Al-N compd., as that the resulting Al compd. remains wholly or in part undissolved. The undissolved Al compd. can be brought into soln. either by bases or by acid, and sepd. from the undecomposed admixts. of the initial materials. The resulting crude reaction mass serves also especially for the production of Al double salts, for which purpose the addition of acid or acid salts and any still lacking alkali or alk. compd. only is necessary.

Ger., 235,868, Aug. 7, 1909. Idem. In the manuf. of ammonium salts from nitrogen compds. of Al, the latter can be decomposed to yield NH<sub>4</sub> salts or their solns, and free Al<sub>2</sub>O<sub>3</sub> which can be sepd. and re-used by treating the Al-N compds. in aq. suspension, preferably at a raised temp., with an acid or an acid salt or a salt yielding acid in the necessary amt., or slightly in excess thereof, for the conversion of the binding N into NH<sub>4</sub> salt, or by employing an acid, such as CO<sub>3</sub>, which does not form a stable salt with Al. The reaction proceeds in the sense of the following equations:  $2AIN + 2HR + 6H_2O = 2AI(OH)_3 + 2NH_4R$  or  $6AIN + Al_2(SO_4)_3 + 24H_3O = 8AI(OH)_3 + 3(NH<sub>4</sub>)2SO<sub>4</sub>.$ 

Ger., 235,800, May 5, 1909. RENÉ MORITZ, Wasquehal, France. Construction of the lead chamber for the manufacture of sulfuric acid, whereby the supports are omitted, suspension being substituted therefor. Details of the construction are specified.

Ger., 235,835, June 4, 1909. Addition to 171,991, Apr. 2, 1905; cf. Chem. Zentr., 1906, 379. BAD ANILIN- U SODA-FABR., Ludwigshafen a/Rh. Mfg. stable anhydrous hyposulfite from NaHSO<sub>3</sub> and formaldehyde Na sulfoxylate, by mixing conc. solns. of the initial materials in the presence of NaCl at a temp. above 50°.

Ger., 235,954, May 24, 1908. International Salt Co., London. In the refinement of salt, the initial material is placed, in hollow heaps, on a hearth, and fused by means of hot combustion gases, whereupon the molten salt flows down into a collecting chamber beneath the hearth, where it is protected from the hot gases and remains in the fused state.

Ger., 236,044, Jan. 14, 1910. O. SERPEK, Niedermorschweiler bei Dornach. In the conversion of difficultly reducible oxides into other compounds, especially nitrides, with direct application of all the necessary heat by means of hot gases, all the heat is brought to bear either on the oxide alone or on the oxide and the reducing agent, or upon both, separately. In this way the oxides, by the employment of the most suitable excess of O, can be heated to the highest possible temp., and pure O may be employed to burn the heating agent. The process is especially adapted to the production of AlN from Al<sub>2</sub>O<sub>3</sub>.

# 10. GLASS AND CERAMICS.

G. E. BARTON, A. V. BLEININGER.

Cameos of Pressed Glass. Anon. Diamant, 33, 655-6. J. B. PATCH.

The Drawing and Blowing of Hollow Glass Articles. Anon. Glashatte, 41, 668-9.

J. B. Patch.

Green Enamels for Galvanized Iron. Anon. Glashatte, 41, 604-6, 649-51.—The writer gives general directions for enamelling and the following formulas: For the first layer and enamel, SiO<sub>2</sub> 500, borax 375, feldspar 330, MgO 200. If it is desired to lower the m. p. the feldspar content may be increased. The enamel may be colored green by adding a thoro mixture of smalt 125 and Naples yellow 100. The latter is prepared by roasting Sb<sub>2</sub>O<sub>4</sub> 100 and PbO 75. A dark green is produced by CoO 100, Naples yellow 50, MnO<sub>2</sub> 10-20. Nankin yellow may be used to vary the green shade, as for example, CoO 100, Nankin yellow 40, Naples yellow 35. To obtain a light yellow-green, Cr<sub>2</sub>O<sub>2</sub> 50 and Naples yellow 20-35 may be used. A pleasing green is also given by the addition of 1 pt. or slightly less of CuO to 5 pts. of the enamel mixture. Small amts. of Fe oxide may be added to brighten the green.

J. B. Patch.

The Enamel Industry. R. Vondracek. Sprechsaal, 44, 264-7; thru Chem. Zentr., 1911, I, 1896.—A report on its condition in 1910. E. J. Crane.

Colloid Chemistry in Pottery Manufacture. Anon. Pottery Gaz., 36, 800-2.

—Brief review of colloids with reference to ceramics. Considerable attention is given to work on plasticity of Rohland, Acheson and Ashley (see C. A., 1, 90; 5, 173 and Trans. Am. Ceram. Soc., 6, 31).

H. K. CUMBINGS.

Color Standardization. J. W. LOVIBOND. Pottery Gas., 36, 802-4.—Continuation of C. A., 5, 227 with demonstration and further description of the "tintometer" and "chromo-pyrometer" there discussed.

H. K. C.

Carriers' Receipes for Porcelain Body and Glazes. Anon. Pottery Gas., 36, 797-8.—Translation of 24 receipes as reconstructed by Auclair in Sprecksaal.

KC

Researches on the Burning of Porcelain. M. LARCHEVEQUE AND C. NOBLE. Ctramique, 14, 122-5.—From various expts. the authors conclude that F is present in appreciable amts. in the glaze and paste of hard porcelain, that the elimination of fluorides is aided by the presence of H<sub>2</sub>O and that the fault known as grease-spot or "ressui" is due to fluorides.

H. K. Cumbings.

Vencalsic Process for Regulating and Increasing the Output of Ceramic Plants. L. Dorian. Céramique, 14, 125-9.—The average heat loss in a brick or tile plant is analyzed as follows: Of the heat produced in the kiln, 49% (20% carried away in the fired ware, 20% radiated, 9% lost in smoke); of the heat produced in the steam power plant, 52% (40% in escaping steam, 12% in smoke). These losses may largely be eliminated, thus increasing the output and rendering the plant independent of weather conditions (moisture and draft), without rebuilding the establishment, by adding the "Vencalsic group." The latter consists of 4 units: a regenerator which is supposed to utilize all waste heat in warming the air for the driver, a special furnace to meet demands for excess heat in any part of the plant, a ventilating system for collecting the waste heat and supplying warmed air to the driers, an automatic motor (running day and night) to drive the fans. The daily operating expense varies from \$1 to \$3 with the size of the plant; in the case of a plant producing 5 million brick annually the output was exactly doubled by this installation. H. K. Cumings.

Preparation of the Black Enamel of Greek Pottery from Natural Magnetic Oxide of Iron. L. Franchet. Compt. rend., 152, 1097; Céramique, 14, 150-1.—Brongniart (1844) following Salvétat called attention to the presence of some MnO in the black enamel of the Graeco-Italian pottery but the presence of Mn is accidental and not necessary to the color which is produced by Fe<sub>2</sub>O<sub>4</sub>. Verneuil suggested that Fe was introduced as a metallic powder, his hypothesis being that the Greeks obtained this by reducing the oxide with C. The black enamel consists of a frit and a coloring agent; the opacity being due to an undissolved excess of the latter. The initial frit (18th Dynasty in Egypt) consisted originally of SiO, and salts of K and Na. This frit with the addition of 50% hammer scale was used in making black glass. Hammer scale could not have been used for this enamel which vitrifies above its m. p. and was fired oxidizing. The following formula gave, in the oxidizing fire, a very good imitation of the enamel in question and vitrified about 850° Frit: Quartz sand 55, Na<sub>2</sub>CO<sub>2</sub> 45; enamel: frit 50, magnetite 50. Substituting chalk for 10 parts of SiO, gave equally good results but the use of pure Fe<sub>2</sub>O<sub>4</sub> was followed by oxidation. Glue of some sort was probably used to thicken the enamel and its C on burning would help prevent oxidation. Egypt, not Greece, should have the credit for originating this enamel.

H. K. CUMMINOS.

Tournay Soft Porcelain. II, III. ALBERT GRANGER. Céramique, 14, 144-7, 2 illus.; cf. C. A., 5, 2540.—Manufacture: Tournay was a fritted porcelain. Milet reports that in 1862 the paste was composed of 60% frit while the remainder was Lille

chalk and Tournay marl. This seems to contradict Brongniart and Soil who earlier stated the comp. as frit, clay and marl but this discrepency may easily be due to changes in the character of the frit (sand + grog + Alicant soda) since the "soda" used seldom earried over 25% Na<sub>2</sub>CO<sub>2</sub> and often had 60% CaCO<sub>4</sub>. Casting and pressing into molds were both employed. Tournay porcelain seems to have been less delicate than Sèvres as less pains were taken in setting. Saggers of marl as well as those of fire clay were used. The kiln was cylindrical: 9 ft. high and the same in diam. A fritted glaze (alkaline Pb borosilicate) was applied to the biscuit ware by dipping and underglaze decoration in blue was common.

Early Persian Pottery. Anon. Pottery Gas., 36, 788, 2 illus.—Description of a collection covering the period 700-1326, A. D. discovered in recent excavations in Central Persia and exhibited in London.

H. K. Cummings.

Ceramic Chemical Development of Architectural Terra Cotta. Herman A. Plusch. Brickbuilder (U. S.), 1911; thru Brit. Clayworker, 20; The Brickbuilder, 1911, 53-4.

C. H. Kerr.

Impermeable Roofing Tiles. Anon. Brit. Clayworker, 20, 104.—Burning to vitrification is usually impracticable because of warping. Glazing and tar-coating (tar heated to 100°) are discussed.

C. H. Kerr.

The Press Cylinder. Anon. Tonind.-Ztg., 35, 1067-9.—Objection is raised to the use of conical auger presses, which require considerably more power than cylindrical ones.

A. V. Bleininger.

Copper Red under the Glaze. Anon. Keram. Rundschau, 19, 309-11.—Review of the work of Seger and Vogt.

A. V. Bleininger.

Contribution to the Lead Question. W. PURALL. Keram. Rundschau, 19, 331-3—Many Pb glazes give off Pb to dil. acid solns. due to the fact that they contain either fine metallic Pb globules or a surface film higher in PbO than the glaze layer in contact with the body. This is due to the alternating oxidizing and reducing conditions of the kiln. For acid-proof glazes, the comp. of the glaze is not an all-im\_ortant factor; there are necessary: Proper kilns, long firing, the rejection of very liquid glazes and avoidance of too heavy an application; avoidance of heavy reduction; good draft; slow cooling by clean air; protection of ware from direct contact with fire gases.

A. V. BLEININGER.

Color Standardization (LOVIBOND). 1.

# 20. CEMENT, AND OTHER BUILDING MATERIALS.

C. N. WILEY.

Causes for the Changes in Speed of Hydration of Cement. P. ROHLAND. Stuttgart. Z. Chem. Ind. Kolloide, 8, 251-3.—The speed of hydration of cement is affected by the presence of electrolytes either dry or in soln. The effect is greater if the electrolyte is dissolved and is approximately proportional to the amt. of electrolyte present. Na<sub>2</sub>CO<sub>3</sub>, AlCl<sub>2</sub>, and K<sub>2</sub>S aid the hydration; K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>, and sulfates, with the exception of Al<sub>2</sub>(SO<sub>4</sub>)<sub>2</sub> and the alums hinder the hydration. Part of the process of setting depends on the coagulation of colloidal hydroxides of Si, Al, and Fe, which process is effected by electrolytes. A high temp. also favors the hydration of the cement.

H. ISHAM.

Relation of the Lime Content of Cement to Durability of Concrete. H. S. SPACK-MAN. Concrete, 11, 52-3.—Quotes from Bied's paper (C. A., 3, 2866). The disintegrating action upon concrete is caused principally by solns. of Ca and Mg sulfates. As

city ground-waters usually contain H<sub>2</sub>SO<sub>4</sub> a cement containing much lime would form CaSO<sub>4</sub> which, in soln., would decompose the concrete. This condition may be avoided by making the cement with a minimum of lime and the concrete impervious to water.

I. W. RIGGS.

The Manufacture of Portland Cement from Blast Furnace Slag. R. S. Hury. Chem. Eng., 14, 324-6.—A description of plants 3, 4 and 6, Universal Port. Ct. Co., Buffington, Ind.

A. J. Phillips.

Blast Furnace Stag as Concrete Aggregate. Anon. Eng. News, 66, 185-6.—
The results of compression tests on concretes made from river sands, stags and limestone showed: (1) Bank stags tested higher than either gravel or limestone. (2)
All stag aggregates compare favorably with the gravel standard. (3) Machine stag is
recommended for lighter work where max. strength is not essential. (4) The bank
stag screenings which developed very high strengths is air-cooled blast furnace stag
excavated from waste banks in which it had cooled slowly from the molten condition
while machine stag is made by running molten stag into shallow Fe pans on a conveyor
and wetting it just before dumping.

A. J. Phillips.

Concrete Test Specimens in Sea Water at Charlestown Navy Yard. M. C. TUTTLE. Eng. Record, 64, 229-30.—Photographs are shown of 24 test specimens placed in sea water early in 1909. Their position was such that the tops were seldom immersed while the bottoms were never dry. Of the 4 specimens which have been most affected, 3 were very dry mixes of the same kind of cement while the 4th was a wet mixt. containing soap and alum, the latter having been mixed with the cement while the soap was dissolved in water.

A. J. Phillips.

Practical Tests of Sand and Gravel Proposed for Use in Concrete. R. S. Greenman. Eng. Record, 64, 66–7.—A series of 7- and 28-day mortar, void and loam tests were made on 34 sands according to the methods given in C. A., 2, 1606. The highest tensil strengths were found with the lowest % of voids and the highest % of loam. The smallest % of voids was given by sands having the best grading of the sand grains (see C. A., 5, 2319) and sands with the largest % of fine grains are the poorest. Loam in small amts. is beneficial. Void and loam tests are recommended for field tests.

A. J. PHILLIPS.

A British Example of Electrolytic Corrosion of Steel in Reinforced Concrete. Anon. Eng. News, 66, 207.—Analyses of two rust samples from the steel reinforcing of docks in the harbor of Southampton showed Fe<sub>2</sub>O<sub>2</sub> 79.01-81.01% and Fe<sub>2</sub>O<sub>4</sub> 3.37-4.47%. The presence of the black magnetic oxide is evidence that the rusting is due to electrolytic action (cf. C. A., 5, 2421).

A. J. Phillips.

The Expansion and Contraction of Concrete while Hardening. A. T. GOLDBECK. Eng. Record, 64, 73.—Wet and dry mixes, variously proportional, were either allowed to dry out or kept wet for 15 dys. In the former case, shrinkage began at once; with the very wet mixes a slight expansion for a few days was noted. Assuming that the value of the modulus of elasticity of concrete in tension is 2,000,000 lbs. per sq. in., if the ends of a concrete construction were confined the tensil stress produced by shrinkage should equal 1,000 lbs. per sq. in. and shrinkage cracks should develop at once. With the specimens kept wet the expansion was quite small corresponding to a compression of 200 lbs. per sq. in. The indications are that dry mixes are more sensitive to expansion than wet mixes, and that the effect of moistening concrete for the first few days is to delay shrinkage.

A. J. Prillips.

Proposed Standard Specifications for Steel Bars for Concrete Reinforcement. Anon. Eng. News, 66, 73.—Presented before the meeting Am. Soc. Testing Materials. 1911.

	Structural steel grade.		Hard grade.		Cold
Properties.	Plain bars.	Deformed bars.	Plain bars.	Deformed bars.	twisted bars.
Pmax. Bessemer	0.10%	0.10	0.10	0.10	0.10
Open-hearth	0.05	0.05	0.05	0.05	0.05
Ult. tensil strength, lbs. per					
sq. in	55,000-70,000	55,000-70,000	80,000 min.	80,000 min.	55,000
Yield point, min., lbs. per					
sq. in	33,000	33,000	50,000	50,000	50%
Elongation, min. % in					
8 in	1,400,000	1,250,000	1,200,000	1,000,000	
	Tensil str.	Tensil str.	Tensil str.	Tensil str.	

Check analyses by purchasers permit an excess of 25% P above the required limits.

A. I. Phillips.

Road Tests of Oil Cement Concrete of the U.S. Office of Public Roads. Anon. Eng. News, 66, 74-6.—Circ. No. 94, Office of Public Roads. A progress report. A. J. P.

New Maryland Cement Plant. Anon. Eng. Min. J., 92, 390.—Description of the plant of the Tidewater Port. Cement Co.

A. J. PHILLIPS.

Recommended Specifications for Bituminous Materials. T. W. Allen. Eng. Record, 64, 200-1.—The N. Y. State Highway Comm. recommends the following spec. for a climate similar to that of N. Y. The asphaltic bituminous material: (1) Shall be free from water or decomp. product; (2) no oily or granular constituents shall be present; (3) sp. gr. at 25° not less than 13° Bé.; (4) penetration between 14 and 18 mm. when tested for 5 sec. at 25° with a No. 2 needle weighted with 100 g.; (5) loss in wt. not over 4% when 20 g. in a cylindrical vessel (21/2 in. diam. by 3/4 in. height) are held at 163° for 5 hrs., not more than 8% when held 5 hrs. at 215.5°; (6) residue at 25° smooth and hard yet readily dented by the finger; (7) solubility at air temp. in CS, at least 99.5% for residuum or gilsonite products, 96% for Bermudez, 81% for Cuban and 66% for Trinidad which are not residuum; (8) solubility at air temp. in 76% Bé. petroleum distilling between 140 and 190° F. shall be 79-84% for residuum, gilsonite and Bermudez products and 72-82% for others; (9) the bitumen shall show 9-15% fixed C; (10) open flash point not less than 176.6°; (11) not more than 4% paraffin scale may be present; (12) ductility not less than 30 cm. at 25°; (13) toughness not less than 30 cm. at o°. Directions are given for making tests No. 12 and 13 which are new and which are especially recommended when the material is to be used as a filler for brick or stone block pavements. A. J. PHILLIPS.

Use of Fluorides for Combating Dry Rot. R. NOWOTNY. Chem. Ztg., 35, 546-7.

—The author discusses the need of preservatives, and summarizes the work done on the fluorides. NaF, ZnF<sub>2</sub>, or (ZnF)<sub>2</sub>O may be used to impregnate timber. These salts have five times the antiseptic strength of CuSO<sub>4</sub> or ZnCl<sub>2</sub>, are cheap, do not discolor wood, are not injurious to health and wood treated with fluorides resists rot longer than with other preservatives.

SIDNEY BORN.

General Directions and Specifications Relating to the Tar Treatment of Roads. The Road Board; thru J. Soc. Chem. Ind., 30, 670.—No. 4, Surface Tarring: The tar is to be derived from the coking of soft coal, with not more than 10% mixt. of tar from carburetted water gas. Sp. gr., 1.16–1.22. H<sub>2</sub>O content less than 1%, with not more than 5 grains NH<sub>4</sub>OH per gal. tar. Shaken 15 min. with 20 vols. H<sub>2</sub>O, at 21° not more than 5 grains per gal. H<sub>2</sub>O of phenoloid compds. (calc. as phenol) shall be extracted. Tar from gas works must not be treated further than for freeing H<sub>2</sub>O or NH<sub>4</sub>OH or light oils. Less than 1% must distil below 170°, and 16–26% from 170–270° (excluding H<sub>2</sub>O). Free C not over 16%. After 30 min. at 30°, distillate must be clear and free from naphthalene and other solids. When distilled to 300°, the

residuum must not be more than 73%. In distillations, the thermometer bulb is placed opposit the side tube of the flask. No. 5, Retarring: Sp. gr. of tar, 1.18-1.24. Other specifications as above except that tar must yield no distillate below 140°, and only 3% up to 220°; be ween 140 and 300°, 15-21%. Free C less than 18%. No. 6, Pitch: Must be derived from coking soft coal, and may be mixed with 10% of carburetted water-gas tar. Not more than 1% is to distil below 270°, from 2-5% between 270 and 315°. Free C not over 22% if not too expensive to prepare; otherwise 28% is allowed with a reduced proportion of sand as filler. Tar oils are to be derived as above. Sp. gr. 1.065-1.075. After 30 min. at 20°, must be clear and free from solids. Not more than 1% of distillate below 140° is allowed, 30-50% between 140-270°. Pitch and tar oils are to be mixed in the proportion of 88-90% pitch to 12-10% of oils. R. E. Humphreys.

Notes on Creosoted Wood Block Pavements. P. E. GREEN. Eng. News, 65, 474-5.—Modern block is impregnated with oil to prevent decay and keep out water. The oil must contain agents to destroy the fungi and bacteria causing dry rot, must be non-drying at ordinary temp. and stable at low temp., and must not have such a high m. p. as to cause the wood to become brittle. Hard dense woods do not wear as well or take treatment so freely as the softer porous timbers such as long-leaf yellow pine, tamarack and southern black gum. Dead oil of coal tar containing naphthalene and anthracene as the principal germicides is considered the best. A. J. Phillips.

Specifications for Wood Block Paving. S. Whinery. Eng. News, 65, 501-2.— A criticism of the Spec. of the Assn. for Standardizing Spec. with regard to the impregnating material recommended (see C. A., 5, 2551-2). The coal tar product recommended, is not a creosote oil and adoption of the spec. would prohibit the use of creosote oil (cf. C. A., 5, 2427, 2428, 2720).

A. J. Phillips.

Recommendations for Use in Chicago Street Paving Specifications. S. WHINERY. Eng. News, 65, 471.—The use of natural cement should not be permitted. For brick paving a grout instead of a bituminous filler should be used and 18 instead of 20% should be the max. loss of wt. allowed in the rattler test. The use of experimental asphalts should be discontinued and more detailed spec. formulated as to the quality of crude asphalt and the methods of refining it. The spec. for creosote oil should be changed in order to prevent exudation of tar in hot weather and sand instead of pitch should be used for joint filling.

A. J. Phillips.

# 21. FUELS, GAS AND COKE.

### I. D. PENNOCK.

Measurement of Heat Flow thru Furnace Walls. W. T. BRAY AND E. KREIS-INGER. Mining Sci., 63, 417-20; Bur. Mines, Bull. 8; Chem. Eng., 14, 374-6; Eng. Record, 64, 279.

J. J. M.

Lignite Briquets for Industrial Purposes. Anon. Iron Coal Trade Rev., 83, 83.—Statistics of the development of the German lignite industry, show that while the production of lignite has increased only very slightly, the sale of briquet has increased 36.6%. Briquet firing finds favor wherever uniformity and intensity of heat and good capacity of regulation are desired, as for drying furnaces, malt kilns, and tar distillation. Briquet-fired producers offer the special advantages of giving a gas of high heating value, uniform comp. and very low S content, with simplicity of handling material and economy of labor.

HORACE C. PORTER.

Briqueting Tests of Lignite. C. L. WRIGHT. Dept. of the Interior, Bur. Mines., Bull. 14.—The object of the tests was to det.: (1) The possibility of briqueting Am.

lignites without adding binder to them; (2) the suitability of German Brown Coal briquet presses for briqueting Am. lingites; (3) the % of moisture needed in the briquet material to give best results; (4) approx. com. cost of briqueting lignites; (5) the weathering qualities of briquets as qualities compared with raw lignites. Lignites containing less than 1.4% of matter sol. in CS<sub>2</sub> (calc. to H<sub>2</sub>O free basis) have not been briqueted with the German machine nor any other machine without the addition of binder. Lignites containing 1.4–1.5% matter sol. in CS<sub>2</sub> are difficult to briquet, but lignites containing more than 1.5% matter sol. in CS<sub>3</sub> were briqueted with German machine without addition of binder. The % of H<sub>2</sub>O that dried lignite must contain to give good briquetes with German machine is proportional to matter sol. in CS<sub>3</sub>, since, if two lignites have same ash content the one richer in bitumen may be dried more and will give briquets of higher heat value. The author emphasizes the value of the CS<sub>2</sub> ext. test on lignites as a reliable indicator of their briqueting qualities and states that lignites sol. 1.5% in CS<sub>3</sub> or more may be briqueted in German type machine without the addition of artificial binder.

C. A. COLE.

The Question of the Spontaneous Ignition of Lignite Briquets. F. W. HINRICH-SEN AND S. TACZAK. Mitt. Königl. Material prüfungsamt, 29, 220-47.-When piled in the open air, briquets showed no appreciable rize in temp. over the surrounding air, nor did they when piled in a dry closed space with all precautions, such as ventilating shafts, even at a temp. of 40°. Storing in a closed space, with daily addition of water was also without bad effect. Parallel expts. with and without ventilating shafts in the piles showed no appreciab e difference. More work was done on fresh briquets. Results showed that whereas no elevation of temp. took place in piles provided with shafts and kept in a closed dry space, when the briquets were piled close together the temp. within the pile rose to 60°, gradually falling after the max. was reached. Results did not vary essentially when the heaps were kept moist. Comparative expts. with closely piled heaps and dumped heaps of briquets in closed spaces showed that the latter had no tendency to become heated under the same conditions that caused a rize in temp. in the former. It was concluded that when ordinary precautions are taken and piles are not more than 4 meters high the storage of lignite briquets in closed spaces is attended with no danger. Numerous tables, curves, and diagrams are given. M. HEIDELBERGER.

A Commercial Fuel Briquet Plant. Discussion of Blauvelt's Paper. R. Schorr. Bull. Am. Inst. Mining Eng., 47, 961-3; cf. C. A., 4, 2996.

J. J. M.

Philippine Firewood. A. J. Cox. Philipp. J. Sci., 6, 1-19.—Fuel values for various woods are given.

J. J. M.

Safety Lamp Test. Anon. Prog. Age, 29, 608.—A safety lamp for use in mines based on Davy's principle, modified by German mfgrs. When the above lamp was lighted and a stream of coal gas played around it, the gas ignited inside the lamp but not outside. Placed in a chamber containing an explosive gas the lamp gradually went out. Tests to det. the reliability of the lamp under rough conditions indicate that it is a valuable lamp.

C. A. COLE.

Filament Ignition of Gas. Howard Lyon. Am. Gas Light. J., 95, 60.—A Pt alloy filament gives higher m. p. than Pt, shows greater chem. inertness to gas impurities and greater catalytic power, enabling the filament to develop the needed temp. with a minimum of applied heat. Catalytic action starts at about 500° but the temp. rizes much higher, to 1500° and even to 2000°. By proper control of the amt. of gas reaching the filament the temp. of the filament is easily controlled. The filament is heated by the passage of current from 1 dry cell which should give 100,000 ignitions in its life.

M. B. Smith.

Pathogenic Properties of the Gases Discharged by the Exhaust Pipe of Gas Engins. S. Delépine. Public Health, 1911, 309–14; J. Soc. Chem. Ind., 30, 674.—The exhaust gases when the engin was fully loaded, are dangerous because high in CO<sub>2</sub> and containing no oxygen. Insufficient air supply gives an exhaust containing 4.5% CO; dangerous unless diluted with 50 volumes of air. Discharge of exhaust gases into sewers should be forbidden and discharging into any confined space accessible to persons should be avoided. Mice and guinea pigs were used to study the pathogenic properties of the gases.

W. H. McLauchlan.

Gas and Smoke. H. RIDDELL. J. Gas Lighting, 115, 294.—A discussion as to the relative merit of gas and coal for domestic heating; quickness of action and regularity is claimed for gas altho the ultimate cost in Belfast in comparative economy is lower for coal. Flue gas from coal fire showed per million cu. ft. gas, 13.58 lbs. C, with 5.4 lbs. of unconsumed hydrocarbons; 24.17 parts per 100,000 CO, 1.36 parts SO<sub>2</sub> and some NH<sub>2</sub>. Flue gases from gas fire gave no solid matter and only traces of other impurities. Purity of air, ventilation and humidity are maintained better with gas than with coal under same conditions. High pressure gas gives very economical results for furnace heating; 100 in. pressure for 2600° F.; 70 in. for 2000° F.; 35 in. for 1000° F. At 1 s. 3 d. per 1000 cu. ft. gas is as cheap as coke at 15 s. per ton. M. B. SMITH.

Escape of Gas from Coal. H. C. PORTER AND F. K. OVITZ. Dept. Interior, Bur. Mines, Tech. Paper No. 2.—The results of the expts. show that certain Am. coals whose mining is attended with danger from accumulation of inflammable gas, liberate this gas not only while they are being broken down in the mine, but also during a long period thereafter. At first the gas escapes rapidly, but the rate diminishes and tends toward final cessation in 3–18 months. If the vol. of the small lumps of coal used in the expts. be taken as the unit of measurement, about  $\frac{1}{4}$  vol. of methane escapes during the crushing of the coal, and  $\frac{1}{2}-1\frac{1}{2}$  vol. on continual exposure to the air. The loss of fuel value by the loss of gas is small, but the danger of accumulation of explosive gas from this source in the mines and coal bunkers is sufficient to justify its being taken into account in the ventilation of mines and in the storage of coal.

C. A. COLE.

Apparatus and Methods for the Sampling and Analysis of Furnace Gases. C. J. W. Frazer and E. J. Hoffman. Dept. of Interior, Bur. of Mines, Bull. 12; Min. Sci., 64, 57-9.—A paper descriptive of methods and app. used where the detection and detn. of a gas in small quantities is accurately reported. C. A. COLE.

Helium and Argon in the Natural Gas of Neuengamme. A. VOLLER AND B. WALTER. Hamburger Wiss. Insts., 28, (1910); thru Petroleum, 6, 1062.—The authors failed to observe the He line, as has been stated, in the spectrum of the light emitted by the burning of the natural gas. Argon and He, however, as well as the yellow line at 5876, and the green line at 5016, were observed after the dispersion, by means of the Mg electrode, of the N spectrum. The He content amounted to 0.01-0.02%, and the A to 0.05% of the natural gas.

O. E. Bransky.

Natural Gas in the Tertiary Basins of Transylvania. J. SZADECZKY. Petrolsum, 6, 773.—The gas consists of 99.25% CH<sub>4</sub> and 0.75% N. Over 900,000 cu. m. issue daily from one well. The gas occurs in miocene formations, and seems to extend along partly broken anticlines. The latter are undulatory; the upper strata are often almost horizontal, but the lower the strata the greater the undulation.

O. E. B.

Cokes and Oils for Water Gas Manufacturing. J. H. WARNICK. Am. Gas Light J., 95, 21.—A discussion as to the constituents and properties of a good gas coke and the bearing of these properties on a minimum cost and best machine results, the source of supply and oils available for water gas mfgrs. Coke: Best

generation fuel has from 21-32 times as much fixed C as volatil C; ash should have high fusing pt., should show about equal amts. of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>, a minimum amt. of Ca and lowest possible amt. of Fe, Mg and alkalies. An undesirable ash shows: 30% SiO<sub>2</sub>; Fe, about 15%; Ca, above 10%; and alkalies + Mg, above 3%. Moistu e in coke charged, about 4%. S should not go over 1.1%. Analyses are given of 11 typical cokes, the best cokes coming from W. Va. and Penn. Oils: The greater the % of paraffins the better the oil. S is a detriment. Analyses of oils from 10 oil fields are given. Oils from the Appalachian fields consist mainly of paraffines with some benzine; Ohio and Indiana oils of paraffins but contain more of benzol series; California oils chiefly benzols with no paraffins; Texas oils, largely of the  $C_nH_{2m-2}$  and  $C_nH_{2m-4}$  series with little paraffins or benzols; Kansas light oils are largely paraffins; the heavy oils contain only a small amt. of paraffins. Louisiana oils are similar to Texas oils.

M. B. SMITH.

Light Oil and Fuel Oil (LE CONTE). 22.

Ger., 236,159, Nov. 15, 1910. R. PAWLIKOWSKI, Görlitz. Mfg. incandescent bodies from artificial silk by winding up untwisted threads from artificial silk solns. which have not yet been impregnated.

# 22. PETROLEUM, ASPHALT, COAL TAR AND WOOD PRODUCTS.

#### R. E. HUMPHREYS.

Petroleum Industry in 1910. RICHARD KISSLING. Chem. Ztg., 35, 861-2, 878-9.

J. J. M.

History of Petroleum. XI. Anon. Petroleum, 6, 1053-6.—The visit of Engelbert Kampfer to Baku in 1684, and his account of the oil fires, the so-called eternal fire, are described.

O. E. Bransky.

Lubricating Oil. F. H. Mann. J. Eng. Soc. Penn., 3, 227-54.—A general discourse on the physical and chem. properties of lubricating oils in which is given the following "Franklin" Free Carbon Test: A 2-oz. beaker, ½ filled with oil, is placed in a shallow sand bath over a bunsen burner. The bulb of a thermometer is immersed in the oil and the latter heated up to 315.5° in ½ hr., held at this temp. for ½ hr. and allowed to cool to ordinary temp. Ten cc. of oil is then mixed with 190 cc. of 88° gasoline and filtered at once through a folder filter. Wash with 100 cc. gasoline, wet the residue with 25 cc. CHCl<sub>2</sub>, and finish washing with gasoline. The filter is dried in air and compared with carbon deposits of other oils.

A. J. Philllips.

The Relative Value of Light Oil as Compared with Fuel Oil. J. N. LECONTE. J. Am. Soc. Mech. Eng., 33, 876–9.—Oils rich in H are of light d. The range of Calif. oils is from unity to 0.84, or 10–38° Bé. The majority of fuel oils range from 10–23° Bé. The presence of impurities, such as  $H_2O$ , N and S, destroys any exact relation between heat units per lb. and d. The results of detns. of heating value and d. on a no. of oils made in lab. of Univ. of Calif. show that the heating value per lb. of crude oil increases as d. diminishes, but it does not increase as rapidly as the wt. per unit of vol. decreases. The heating value per barrel of heavier oil is therefore greater than that of light oil.

A. J. Phillips.

The Optical Relations and other Properties of Japanese Petroleum. M. RAKUZIN. Petroleum, 6, 1048-53.—The author cites the work of Takano for the geographical, geological, chem. and physical relations of Japanese oil. The oil occurs mostly in the tertiary formation. All the hydrocarbons obtained from different crude oils be-

long to the methylene series or to the naphthenes of the ethylene group. The heavier oils contain naphthenes with 2 or more rings. Oil from Amase and Ilirei contain solid paraffin. All the oils, except those from Kusodsu and Koguchi, contain 1-40% of aromatic hydrocarbons. They resemble in chem. comp. Russian and California oils. Japanese oils of the same sp. gr. as American oils boil at a lower temp. than the latter. The I values vary from 0 to 9.79; the expansion coeffs. from 0.000825 to 0.000615. The heat value is generally high (Miyagawa oil 11,636 cals.). Of 10 oils, only 1 oil (from Amaze) was inactive. This oil was obtained at a depth of 2100 ft. Seven oils were polarimetrically semi-transparent; K (in  $C_0H_0$  soln.) from 1.5 to 15.75%. Two oils were non-transparent; K 1/4-1/2%. Dextrorotation in a 200 mm. tube was observed in the distillates from Urace oil, and in oil from Amaze. O. E. Bransky.

Report of the Committee D<sub>2</sub> upon Lubricating Oils. Drugs, Oils and Paints, (26), 1910, Nos. 6 and 7; thru Petroleum, 6, 2029–31.—Committee D<sub>2</sub> of the Amer. Soc. for Testing Materials, after comparing the Saybolt, Redwood and Engler viscosimeters, recommends that the viscosities of oils and similar substances be detd. by the Engler app., and that the results be expressed upon the basis of water. Numerous tables and curves are given.

O. E. Bransky.

Petroleum Deposits in the Bavarian Tyrol. Board of Trade J., June 1, 1911.—Found principally on the western shore of Lake Tegernsee, 35 miles south of Munich. 304 metric tons were produced in 1909. The crude is equal to the best Pennsylvania oil as regards its content of naphtha and paraffin wax. The oil-bearing strata have been traced for several miles along the northern flank of the mountains of the Bavarian Tyrol.

R. E. H.

Concrete for Oil Reservoirs. R. J. AREY. Eng. Record, 64, 272.—Concrete linings are reported successful in the Southwest with oils of asphaltum base. In some cases the oil penetrates concrete 11/2 in. However, the Standard Oil Co. has discontinued the use of concrete linings for oil and distillate. LANGDON PEARSE.

The Charige in the Asphalt Content of Dark Petroleum. G. MEYERHEIM. Mitt. kgl. Material prasums, 29, 184; thru Chem. Zentr., 1911, II, 61; cf. C. A., 4, 2871.

—After 6 months' standing and again at the end of 8 months an increase in the asphalt content of 2 dark petroleums was observed; Ra rays seemed to have no influence on this change.

E. J. Crane.

Technical Analysis of Petroleum Asphalt. D Lohmann. Chem. Zig., 35, 357; thru Petroleum, 6, 2076; cf. C. A., 5, 2174.—The following tests are made: penetration at 32°, 77°, and at 100° F; total bitumen sol. in CS<sub>1</sub>; insol. organic matter; mineral matter; bitumen sol. in CCl<sub>4</sub>, and in naphtha of 70° Bé.; liquid hydrocarbons; fixed C; ash, S, and ductility before and after addition of flux. The penetration and ductility are important tests in detg. the quality of an asphalt. The fixed C is detd. by the method devized by the Comm. on Coal Analysis of the Amer. Chem. Soc. S is detd. in the Mahler bomb; ductility, by the Dow and Smith app. Petroleum asphalt should represent pure bitumen, almost completely sol. in CS<sub>2</sub>, and, within a few %, in CCl<sub>4</sub>. Its solubility in naphtha of 70° Bé., or in C<sub>6</sub>H<sub>4</sub> (sp. gr. 0.700), indicates the extent of decomp. during the distillation. The insol. C lessens the ductility. The S content seems to have no influence upon the technical use of the asphalt. O. E. Bransky.

United States Asphaltum Production. DAVID T. DAY. Oil, Paint and Drug Rep., June, 12, 1911.—Substances similar to rubber can be prepared from elaterite and tabyite, and when made into automobil tires have good wearing qualities. The most important of these is wiedgerite, a soft, moist material, high in S, resembling liver, and turning almost black when exposed. It may prove valuable in the manuf. of rubber substitutes.

R. E. H.

Coal Tar Products. J. G. O'NEILL. Prog. Age, 29, 660-1.—The only possibility of competing with Germany for our own home market is in a few raw materials, vis., pitch, creosote oil and light oils. We can compete in these materials because scarcely any labor, small installation cost and little skill is required. In purified coal tar products the U. S. has no advantage. The only possible development lies in the recovery of benzene from by-product coke ovens.

C. A. Colb.

Turpentine and Rosin Production in the United States in 1910. U. S. CENSUS BURRAU REPORT. Board of Trade J., June 29, 1911.—27,750,000 gals. (\$17,680,000) turpentine as compared with 29,000,000 gals. (\$12,654,000) in 1909. 953,000,000 lbs. (\$18,255,000) rosin in 1910, as against 914,000,000 lbs. (\$12,577,000) in 1909.

R. E. H.

Methods for the Utilization of Wood Waste. GEORGE WALKER. J. Soc. Chem. Ind., 30, 934-6.—Resinous stump-wood is now destructively distilled or extracted with steam and solvents with profit in western and southern states. Sawmill waste gives a poor yield of AcOH and MeOH, and the residual charcoal is nearly worthless except for fuel, owing to its fine subdivision and distance from market. Turpentine is secured only from the wood of trees that have been boxed for turpentine. On these "box faces" is found considerable hardened oleoresin. Hard wood waste yields much more AcOH and MeOH. For the production of illuminating gas from sawdust, the author describes a successful plant consisting of 2 benches of 5 retorts each. The retorts are cast-iron, 12 ft. long, 12 in. diam., 11/2, in. thick, and provided with conveyor screws. The dry sawdust is fed to the retorts in a thin stream. It carbonizes in its passage and is then delivered into air-tight cars. From the vapor main the gases pass to a condenser and scrubber. The by-products are worthless. Wood waste could be crushed and used in the same way. Dried sawdust pressed into briquets soon absorb water and crumble. In ordinary wood distillation, the production of volatil products ceases at 275°. If the retort is then cooled, the wood is found to be of a dark-red color and contains nearly all the tar. In this form it finds a com. use as kindling wood. The tar produced in wood distillation amts., in the case of hard woods, to 15 gals. per cord. When redistilled with steam, it yields MeOH and considerable light oils. The oil is reddish brown, sp. gr. 0.920-0.960, and when redistilled with steam and treated with NaOH becomes colorless and has a penetrating odor. It is an excellent solvent, especially for pyroxylin, and mixes readily with nearly all oils. When the wood is further distilled without steam it yields 30% of heavy oil, sp. gr. 1.02-1.06, b. p. 200-300°, which may be used directly or mixed with dead oil from coal tar for preserving wood, or t eated with NaOH and H2SO4 for pure wood creosote. The oil toward the end of the distillation contains paraffin. The residuum is useful for producing briquets with 90-92% coal dust. After a pine tree has been killed by "boxing," the oleoresins concentrate at the heart and the bark and sap-wood decay. The heart-wood sometimes contains 40% oleoresin, and when such wood is destructively distilled, it yields oils similar to those produced by like distillation of rosin, together with some AcOH, MeOH and uncondensable gases. If the turpentine of such wood be extracted with steam, the resin can be extracted with volatil solvents. A plant for this purpose in S. Carolina is producing 200 bbls. rosin per week and considerable turpentine. Thus, in 12 hrs., 5 times as much rosin and turpentine is obtained as the same trees would have produced in 4 yrs. by boxing. There is enough of this waste material in the southern states to supply the world in rosin and turpentine for many yrs. The wood, after treatment, is suitable for wood-pulp industries. In this way the annual growth of a pine forest should serve for all these needs without further destruction. When 25 yrs. old, the tree may be felled, then allowed to lie for 5-10 yrs. for the conc. of the oleoresins in the heartwood and the decay of the bark and sap-wood. Such untapped trees, felled close to the ground and treated as above, produce heavily.

R. E. H.

Pine Oil Obtained in the Manufacture of Soda Cellulose. HILDING. Papierfabrikant, 9, (Fest-u. Auslandsheft), 81-2; thru J. Chem. Soc. Ind., 30, 887.—In the digestion of pine wood by the sulfite process, a soap is skimmed off the surface of the black liquors which, after decomp. by acids, yields a thick, dark-brown oil; substances derived from the lignin of the wood are pptd. as a granular sediment. When washed, the oil deposits crystals; when distilled under reduced pressure the distillate contains no phenolic substances, and by Twitchell's method shows 53.1% fatty acids and 46.9% resin acids. The distillate, after being freed from the crystals of resin acids and distilled under 15 mm. pressure showed oleic, palmitic (most abundant in the first fractions) and linolenic acids. Exam. of the resin acids after washing with petrole and crystg. from alc. showed that the product was a mixt.; m. p. 166-180°; acid value, 186.6; and dextrorotatory. The fatty acids exist in the woods as glycerides, and the ether-alc. extract of the sap-wood showed the reactions of glycerol. The decomp. of the fats causes the acrolein odor of wood tar. In pine wood the fats are chiefly present in the sap-wood, the resins in the heart-wood. Fats are present only in the winter, and are converted into starch in the spring. Phytosterol, m. p. 133.5° and sparingly sol. in alc. may be extracted directly from the soap of the digestion liquors. Spruce contains less fats than pine, it also contains phytosterol. Extraction of winter-felled pine sap-wood with ether gave 0.3-1 5% fats; phytosterol is not over 0.05%.

R. E. H.

Specifications for Bituminous Materials (ALLEN). 20. Naphthene Acids (ALBRECHT). 27.

## 23. CELLULOSE AND PAPER.

### A. D. LITTLE.

Electrolytic Decomposition of Cellulose. Anon. World's Paper Trade Rev., 56, 290.—By electrolysis of cellulose in a neutral bath of KCl under certain conditions of current, size of bath, etc. (not given), <sup>3</sup>/<sub>2</sub> of the cellulose is dissolved and the remainder, while still possessing a fibrous structure, is sol. in 10% NaOH soln. Under other conditions a milky colloidal soln. may be obtained from which acids and EtOH ppt. a thick jelly. The colloidal substance is sol. in 10% NaOH and strong NH<sub>2</sub>. It appears to be a new oxycellulose.

The Present Status of the Industry in Artificial Plastic Materials. F. J. G. Beltzer. Paris. Z. Chem. Ind. Kolloide, 8, 313-25.—A discussion of the industry including wood pulp, cotton, artificial silk and by-products.

H. ISHAM.

Advances in Paper Testing in the Year 1910. W. HERZBERG. Papier.-Ztg., 36, 2122, 2194, 2258, 2329, 2392. V. Nunez.

Japanese Paper. B. Setlik. Oesterr. Chem. Ztg., 14, 110-2; thru Chem. Zentr., 1911, I, 1660.—The author describes several Japanese papers and gives an exact account of the raw materials, the method of manuf., and the microscopic exam. of the paper. E. J. Crans.

Paper Makers' Clays. J. S. Remington, D. A. Bowack, P. Davidson. Aynsome Tech. Lab., Lancashire, England. J. Ind. Eng. Chem., 3, 550-1.—The suitability of a clay for loading material in paper depends upon its freedom from grit and from color. A mechanical as well as a chem. investigation of the clay is there-

fore necessary, since buyers will demand knowledge of the amt. of grit in the clays, A table of analyses of 20 clays is included with a discussion of the results.

V. NUNEZ.

India Papers. CLAYTON BRADLE AND H. P. STEVENS. Paper Trade J., 51, 42.—A descriptive article which deals with the fiber content, the filling, and sizing of India paper, touching also upon the uses to which this paper is put, as well as the qualities desired in it. G. E. MARION.

How Straw Papers are Made in France. Aug. Abadie. Paper Trade J., 52, 44.—The several kinds are described as follows: Paper made in the Limousin region of France is from straw soaked in milk of lime without addition of rags or size. It has a fine yellow color and natural sizing, being crisp and tenacious. That made in the Southwest is soaked warm, contains an addition of hemp waste and is rosin sized. It is very strong but has not so fine a color as that from Limousin. In the Southeast of France the straw paper makers boil the straw in digesters, then wash and add 15% rags and 10% old papers and size with rosin. Besides the yellows, colored papers are made, also those containing 50% or less of straw mixed with hemp, jute or ground wood. Some cook their straw with sol. salts, which produces a soft and silky paper suitable for packing glass and crystal. G. E. MARION.

Pure Oil from Soda Cellulose (HILDING). 22.

Tar Treatment of Roads. 20.

# 24. EXPLOSIVES.

## C. E. MUNROE.

Nitrocellulose Powder for Naval Purposes. Anon. Engineering, 92, 237-8. A controversial article in advocacy of the superiority of the English nitrocellulosenitroglycerin smokeless powder over the U.S. "single base" nitrocellulose powder and citing the experiences of the Brazilian Navy with cordite powder, and especially the circumstances attending the disaster on the Aquidaban, in 1906, where the powder had been subjected for 8 years to excessively high temps., in support of his thesis.

CHARLES E. MUNROE.

Determination of Nitrogen in Nitrates, Gun Cotton and Nitric Esters. M MAR-QUEYROL AND D. FLORENTIN. Ann. chim. anal., 16, 245-50; cf. C. A., 5, 2050.

F. W. SMITHER.

Accidents in the Use of Explosives. Anon. Eng. Mining J., 92, 267-8.— Statistics published by the British Government show that the majority of accidents caused by explosives accur in the winter months when the explosives are more apt to be frozen. ROBERT KANN.

### 25. DYES AND TEXTIL CHEMISTRY.

### L. A. OLNEY.

Amplification of Rota's Method for the Qualitative Analysis of Artificial Organic Dyes. Josh Prats Aymerich. Anales soc. españ. fis. quim., 9, 174-7.—The dyes of Class I (Rota's classification) are difficult to reduce with SnCl, + HCl and, unless heated sufficiently, are liable to be wrongly placed in Class III or IV; the auramines of Class III are reduced or the soln. decolorized on heating to boiling with HCl and

SnCl<sub>2</sub> for a few mins. and might erroneously be assigned to Class I or II. Because of this possible confusion the author proposes the following modification of Rota's Class I:

Class I:	•					
Azo dyes or hydrazones.	soln. colored: shaken with dil. AcOH gives the orig- inal color = basic dye.	not sulfonated; aminoazo compd.				
	soln. colored: shaken with dil. AcOH the original color is changed = neutral dye.	hydroxyazo compd. without carboxyl.				
The aq. soln. treated with KOH and ex- tracted with Et <sub>2</sub> O gives an ethereal ext. with the follow- ing characteris- tics.	soln. color- less: no change. with AcOH — acid dye.	if extracted by Et <sub>2</sub> O from its dil. AcOH soln. — not sulfonated.  not extracted by Et <sub>2</sub> O from its dil. AcOH soln. — sulfonated,	hydroxyazo co with carboxyl. test here for tartrazine. if not tartra- zine, then = azo dye.	•		
		•		group.		

To test for tartrazine proceed as follows: Introduce a strip of woolen cloth into a soln. of about 0.2 g. of the dye in 200 cc. water with several drops H<sub>2</sub>SO<sub>4</sub>, keeping the soln. at b. p. for 15 min. in a water bath. Wash and dry the strip and use it for the following tests, which are given if the dye is tartrazine: Conc. H<sub>2</sub>SO<sub>4</sub>—the fiber turns darker and the liquid clear yellow; 10% H<sub>2</sub>SO<sub>4</sub>—no change in cloth or soln.; conc. HCl—fiber slightly darker and liquid yellow; HNO<sub>3</sub>—fiber orange-colored and liquid yellow; NH<sub>4</sub> water (sp. gr. 0.91)—fiber quite clear and liquid bright yellow; NaOH (10%)—fiber deep red and liquid bright yellow. The tartrazine soln. is decolorized on heating a few mins. with a bisulfite soln.; the color is restored (within 2 min.) by adding a cold satd. soln. of NH<sub>4</sub> persulfate.

The Progress in the Production of Artificial Organic Dyestuffs in 1910. L. LEH-MANN. Chem. Ind., 34, 280-6, 309-13 and 337-41. E. J. CRANE.

Vat Dyes. ERCOLE MASERA. Ind. chim., 11, 117-20, 133-7, 169-72, 181-6.—An address. Samples of cotton dyed with various vat dyes are included in a supplement to the last instalment of the paper.

CHAS. A. ROUILLER.

The Colorimetric Determination of the Content of Dye Solutions. F. H. EIJDMAN. JR. Farber-Zig., 20, 21-4; thru Chem. Zentr., 1909, I, 1673.—Expts. on the colorimetric detn. of benzopurpurine show that this dye, and also the Na and NH<sub>4</sub> salts, can be accurately detd. colorimetrically if the soln. under investigation, after a preliminary comparison, be brought to about the same conc. as the standard soln. and the definit comparison then made. The presence of Na<sub>2</sub>SO<sub>4</sub> has practically no effect on the accuracy of the detn. and com. benzopurpurine can usually be safely compared with the

pure dye. Benzopurpurine dissolved in hard H<sub>2</sub>O cannot be accurately detd. by comparison with dist. H<sub>2</sub>O solns. E. J. Crane.

Fastness to Washing, Washable Colors and their Testing. P. HEERMANN. Mitt. kgl. Material prufungsamt, 1910 (Supplement I), 1-18; thru J. Soc. Chem. Ind., 30, 20.-White linen garments for use in the tropics are often lined with red-dyed material because of the protection afforded due to the high degree of absorption of the light rays of short wave length. In an effort to obtain a dyed material of desired fastness to washing it was found that Turkey red can be dyed, by the "old" method, not by the "new," so as to satisfy all requirements. The author calls attention to the variation in the methods employed for detg. the fastness of a shade to washing. especially in regard to the strength of the solns, of soap and alkali, and the temp. The nature of the fiber (cotton, wool or silk), the degree of staining of white contiguous fiber, and the degree of coloration of the liquors should be taken into account in the interpretation of results. Lang's method (Farber-Ztg., 14, 269) is criticized on these grounds and the following method proposed: Dyed cotton materials are treated (together with white cotton, wool or silk) at 60° and 100° for 15 min. in a soln. containing 5 g. of Marseilles soap and 3 g. of calcined Na CO, per l. The results are to be classified: I. (1) The shade not affected at 100°; (2) shade not affected at 60°; a slight loss of depth at 100°; (3) shade losses a little at 60°; appreciably at 100°; (4) appreciable loss at 60°; (5) very much affected at 60°; stripped at 100°. II. (1) Does not bleed into white contiguous fiber at 100°; (2) does not bleed at 60°; bleeds a little at 100°; (3) bleeds a little at 60°; very much at 100°; (4) bleeds appreciably at 60°; (5) bleeds strongly at 60°. III. (1) Liquor remains uncolored at 100°; (2) uncolored at 60°; slightly colored at 100°; (3), slightly colored at 60°; strongly at 100°; (4) appreciably colored at 60°; (5) strongly colored at 60°. With respect to dyeings on silk and wool the demands as regards fastness must be less exacting. The fastness properties of individual dyestuffs depend very largely upon the method of dyeing and the preliminary or after-treatment employed. A list of dyestuffs with which shades upon cotton may be dyed fast to washing and one of dyestuffs which give fast dye-E. J. CRANE. ing wool are given.

Amplification of Rota's Method of Qualitative Analysis of Artificial Organic Dyes (AYMERICH). 7.

Trihydroxythioxanthones (Ullmann, Sone). 10.

Quinoids (WILLSTÄTTER). 10.

Ger., 235,599, Mar. 23, 1910. BAD. ANILIN- & SODA-FABR., Ludwigshafen a/Rh. In the production of wool mixtures, the wool, in the form of the fat-contg. fleece coming from the carding-comb, is printed and, after steaming, it is mixed on the carding-machine for slubbing.

Ger., 236,074, Mar. 4, 1910. R. LESSER, Berlin. Dyeing with colors derived from benzo- or  $\alpha$ -naphthoquinone. The condensation products of benzo- or  $\alpha$ -naphthoquinone, their homologs and halogen substitution products with aromatic amines are employed as vat dyes. They are obtained by heating 1 or 2 mols. of the amine with a mol. of the quinone in an indifferent solvent such as alc., HOAc, or nitrobenzene, and with 0 without the addition of a neutralizing or catalytic agent, whereupon, by cooling nearly all the dyestuff crystallizes and seps. out in a state of great purity.

# 26. PIGMENTS, RESINS, VARNISHES AND INDIA RUBBER.

### A. H. SABIN, THEODORE WHITTELSEY.

Tests of Paints on Havre de Grace Bridge, and on Steel Test Fence. COMMITTER D-1. Am. Soc. Test. Mat. Eng. News, 66, 120.—19 paints applied on the bridge Oct. 1906, were marked on a scale of 10, in Apr., 1911; for chalking, 14 paints averaged 8.7 (fairly excellent); 4 paints, 7.4 (fairly good); 1 paint, 4.5 (fair). Checking: 16 paints, 9.7; 2 paints, 6:6; 1 paint, 22 (failed). General condition: 9 paints, 8.6 (very good); 8 paints, 7.5 (good); 1 paint, 5.7; 1 paint, 2.3 (failed). After removing the paint (by NaOH) the metal surfaces in most cases were in perfect condition. Paints on the steel test fence after 33 mts. exposure; the best paints tried had as pigments chromates of Pb and Zn, Pb<sub>2</sub>O<sub>4</sub>, PbSO<sub>4</sub>, Fe<sub>2</sub>O<sub>5</sub>, Fe<sub>2</sub>O<sub>5</sub>, and Prussian blue. The worst were CaCO<sub>2</sub>, CaSO<sub>4</sub>, BaSO<sub>4</sub>, chrome resinate, ZnO, lithopone and ultramarine. A. H. Sabin.

Ships'-bottom Paints. H. WILLIAMS. Proc. U. S. Naval Inst., June, 1911; thru Eng. News, 66, 136.—First coating (anticorrosive) 7.25 gal. alc., 7.9 lbs. shellac resin, 0.6 gal. spirit of turpentine, 0.6 gal. pine-tar oil, 9.5 lbs. Zn dust, 28.5 lbs. ZnO = 10 gal. 2nd coating: (anti-fouling) 6 gal. alc., 13.75 lbs. shellac, 1 gal. pine-tar oil, 1 gal. sp. turp., 13.75 lbs. ZnO, 13.75 lbs. Indian red (Fe<sub>2</sub>O<sub>2</sub>), 4.75 lbs. HgO = 10 gal. Dissolve the shellac in the alc., then add the other liquids; the dry pigments are separately mixed and ground with a small amt. of vehicle, then mixed with the remainder of the latter. Avoid mixt. of H<sub>2</sub>O; the surface to be painted is to be dry and free of loose dirt or grease.

A. H. Sabin.

Caoutchouc Resins. F. W. HINRICHSEN AND J. MARCUSSON. Kgl. Materials-pruefungsamt, Berlin. Z. angew. Chem., 24, 725.—The authors detd. the optical activity of the resins extracted from 26 different species of rubber, with a view to obtaining data which might enable this method to be used for the identification of an unknown rubber. They find that almost all rubber resins are optically active, with the exception of the resins from Hevea rubbers—Para and Ceylon. It is only the unsaponifiable part of the resin that is optically active, and therefore the activity is to be ascribed not to derive. of the rubber but to substances which accompany the rubber in the latex. All the above observations hold for the resins before and after vulcanization.

L. E. Weber.

Influence of Ultraviolet Rays on Caoutchouc. L. RAYBAUD. Compt. rend. soc. biol., 71, 216-8.—Measurements of wave lengths of the light and the intensity of damage done. Red is suggested as a covering to protect caoutchouc from ultraviolet rays of the sun.

I. K. Phelps.

Determination of Lead and Zinc in Rubber Goods. H. KÜHL. Suddeut. Apoth.Ztg., 51, 135-6.—Melt in a porcelain crucible 3-4 g. of Na<sub>2</sub>CO<sub>3</sub> and NaNO<sub>3</sub>, equal parts, and drop into the fused mass 2 g. of the finely cut sample. Heat till fusion is completed, cool, dissolve in hot H<sub>2</sub>O, filter, boil insol. ppt. twice with water and collect Pb and Zn carbonates on the filter paper. Dissolve these in HOAc (in the presence of Sb, Fe, Cu, etc., in dil HCl (1 + 1)) and test for Pb and Zn. Ppt. the Pb as ulfide, filter off, digest with (NH<sub>4</sub>)<sub>2</sub>S at 50-60° for 5 min., and convert the washed ppt. into PbSO<sub>4</sub> for weighing. To the filtrate add an acetate, ppt. as ZnS, convert into chloride, then into carbonate, ignite and weigh as ZnO.

A. VORISEK.

The Tetrabromide Method of Hübener. R. BECKER. Gmmi.-Ztg., 25, 531.—Author questions application of Hübener method as given (C. A., 5, 3172) in presence of factis, gutta-percha and balata.

C. E. Bradley.

Hübener's Tetrabromide Method. G. HÜBENER. Gummi.-Zig., 25, 634.—A reply to Becker's criticism of the author's method (see previous abstract). L. E. W.

Hübener's Tetrabromide Method. R. BECKER. Gummi.-Ztg., 25, 677.—A reply to Hübener's discussion (see previous abstract).

L. E. W.

Concerning the Hübener Tetrabromide Method. Gerhard Hübener. Gummi.-Zig., 25, 751.—A reply to Becker (see previous abstract). C. E. Bradley.

Hydrocarbons of the Butadiene Series and Artificial Caoutchoucs Prepared from Chem. Inst., Univ. Kiel. Ann., 383, 157-227. - Methyltetramethyleneglycol, HOCH, CH, CHMeCH, OH, is prepared by adding di-Et pyrotartrate (propane- $\alpha,\beta$ -dicarboxylate), in abs. alc., to Na, the mixture is shaken vigorously and subsequently heated. After removal of the alc., the solid is dissolved in H<sub>2</sub>O, decomp. by CO<sub>2</sub> and the filtrate conc. at 80°, in a vacuum. Colorless liquid resembling glycerol,  $b_{11}$  124-5°;  $d_{18}^{18}$  0.9954;  $n_{D}^{18}$  1.45173;  $n_{R}^{18}$  1.44925;  $n_{T}^{18}$ 1.46107; MD 28.20; My-Ma 0.656. Yield, 20% of the ester. Diacetyl derivative, C<sub>2</sub>H<sub>16</sub>O<sub>4</sub>, from the glycol and b. Ac<sub>2</sub>O; water-clear, mobil liquid with an odor of mustard,  $b_{17}$  116-7°;  $d_{20}^{20}$  1.0434;  $n_{D}^{20}$  1.42717;  $n_{\alpha}^{20}$  1.42509;  $n_{T}^{20}$  1.43658;  $M_{D}$  46.3;  $M_{T}$ - $\alpha$  1.08. Yield, about 150% of the glycol. Sym.-diphenyldiurethan, C1. H2O4N2, from the glycol and Ph isocyanate; lustrous plates from ligroin (b. p. 70-100°), m. 97°. Yield, 120% of the glycol. Tetramethyldiurethan, C<sub>11</sub>H<sub>20</sub>O<sub>4</sub>N<sub>2</sub>, from the glycol and diphenylcarbamyl chloride, in presence of pyridine; colorless prisms from ligroin, m. 102°. Yield, 180° of the glycol. β-Methyltetramethylene oxide formula (I) below, is prepared

from the glycol and H<sub>2</sub>SO<sub>4</sub> (60%), at 100°; colorless, mobil liquid with a pleasant, strongly ethereal odor, b. 86–7°;  $d_{20}^{20}$  0.8643;  $n_D^{20}$  1.41122;  $n_\pi^{20}$  1.40905;  $n_7^{20}$  1.41979;  $M_D$  24.74;  $M_\gamma$ – $M_\alpha$  0.577. Yield, 40–50% of the glycol.  $\beta$ -Methyltetramethylene bromide, BrCH,CH,CHMeCH,Br, from the glycol and conc. HBr, at 100°; heavy, colorless liquid with a sweet odor,  $b_{11}$  84-6°;  $d_{17}^{17}$  1.6986;  $n_{D}^{17}$  1.51217;  $n_{\alpha}^{17}$  1.50918; n<sub>1</sub><sup>7</sup> 1.52722; M<sub>D</sub> 40.64; M<sub>r</sub>-M<sub>g</sub> 1.19. Yield, 190% of the glycol. On exposure to light the bromide evolves HBr; when distilled with quinoline (3.75 pts.) it gives isoprene containing some dimethylene. Yield, poor. A better method of obtaining isoprene consists in treating this dibromide with Me, N, in abs. alc., at 100°, and shaking the viscid product with moist Ag<sub>2</sub>O, after filtering and conc. under reduced pressure, the liquid is distilled under the ordinary pressure. The resulting Me,N is absorbed by H<sub>2</sub>O and the isoprene purified by dil. H<sub>2</sub>SO<sub>4</sub>, followed by Na. It had the following properties: b.  $36-7^{\circ}$ ;  $i_{D}$  51° 10';  $i_{a}$  51°;  $n_{D}$  1.42267;  $n_{a}$  1.41807;  $i_{r}$  52° 5';  $n_{r}$  1.44340 all at 21°;  $d_{21}^{21}$  0.6804;  $d_{4}^{21}$  0.6793;  $M_{D}$  25.45;  $M_{7}$ – $M_{\alpha}$  1.33;  $\Sigma_{D}$  37.46;  $E\Sigma_{D}$  1.72 (4.8%);  $\Sigma_r - \alpha$  1.958;  $E\Sigma_r - \alpha$  0.572 (41.3%). Yield, 50%. The yield of dimethylethylcarbinol from acetone and EtBr is about 80%. The carbinol is volatil with EtO; with anhydrous C<sub>2</sub>H<sub>2</sub>O<sub>4</sub> (0.5 mol.), or H<sub>2</sub>SO<sub>4</sub> it gives trimethylethylene. Yield, above 70%. In glacial AcOH, Br converts it into the dibromide; b<sub>20</sub> 70°. Yield, of pure compd., about 230% of the hydrocarbon. The preparation of isoprene from trimethylene dibromide is best accomplished by dropping it on well dried soda lime, which has been saturated with CO<sub>2</sub>; the temp. inside the apparatus should be 600°. The decomp. is carried out in a vessel of special design; it is made of Fe or brass and the soda-lime is divided by wire gauze, so as to prevent the CaBr<sub>2</sub>, which is formed, from clogging. Yield, of isoprene, 18-20% of the bromide. Omission of the CO<sub>2</sub> produces an impure product, which probably contains trimethylethylene. When trimethylethylene dibromide is dropped into fused KOH, the product consists of a little hydrocarbon together with an unsaturated, monobromo compd., from which the Br is removed only with difficulty. Butadiene (erythrene) is best prepared, in a state of purity,

by the exhaustive methylation of N-methylpyrrolidone (Ciamician and Magnaghi). The process described in Ger. Pat. 28,390 IV., 39, starting from p-cresol, did not give good yields under laboratory conditions. The best results were obtained as follows: com. methylethyl ketone, in Et<sub>2</sub>O, was reduced by H<sub>2</sub>O and Na. The resulting sec. butyl alc. is volatil with Et,O. Yield, 73%. It is converted into butylene by heating with P<sub>2</sub>O<sub>10</sub>. Yield, about 47% of the alc. The use of H<sub>2</sub>SO<sub>4</sub>, or of C<sub>2</sub>H<sub>2</sub>O<sub>4</sub> gives a less pure product. With well cooled CHCl, and Br, it forms butylene bromide, b, 62°. Yield, about 120% of the butylene. It was converted into butadiene by means of soda-lime, in the manner described above. The product is purified by conversion into the tetrabromide, by means of Br, in CHCla, at the temp. of ice + NaCl. Yield, 22%. β,7-Dimethylbutadiene, H<sub>2</sub>C: CMeCMe: CH<sub>2</sub>, is best prepared by converting pinacone into the hydrochloride and decomposing this by means of soda-lime, in the manner described above. The product b. 71°; d<sub>4</sub><sup>16·8</sup>•0.7304; n<sub>D</sub> 1.44321; n<sub>a</sub> 1.43870;  $m_{\tau}$  1.46303, all at 16.5°;  $M_D$  29.80;  $M_{\tau}$ - $M_{\pi}$  1.41. The best method of comparing natural and artificial caoutchoucs consists in an examination of the ozonides and of their decomposition products. The other possible derivatives are the nitrosites and the bromides, which are amorphous, have no definit m. ps. and can only be purified with great difficulty, if at all. Isoprene is converted into isoprene caoutchouc,  $(C_{10}H_{10})x$ , by heating under pressure, with an equal vol. of glacial AcOH, on a H<sub>2</sub>O bath, during 8 dys. The yield varies widely and it is but little, if at all affected by marked variation in the proportion of AcOH, or by changes in the temp. (95-120°). When only a few drops of AcOH are employed, it is necessary to distil the product under the ordinary pressure to remove isoprene and its dimol. polymerization compd., then at 105° (10 mm.) to remove an oily material. The residue is transparent, almost colorless and closely resembles natural caoutchouc. When recently prepared it is readily sol. in ordinary organic media, but this property gradually changes and finally the material becomes insol. After repeated pptn., natural caoutchouc exists in 3 modifications, the (a) form is represented by ordinary, solid caoutchouc, the (b) form is quite insol. and the (c) modification is oily. All are mutually transformable, (c) into (a) slowly and spontaneously; (a) into (c) by warming in soln.; (a) into (b) slowly and spontaneously; (b) into (a), or (c) by b. with AcOH, or Ac<sub>2</sub>O. Similar modifications are observed in the case of artificial caoutchouc, the purer it is the more easily does it pass into the (b) form, but the reverse transformation has not been realized. The best yield of artificial caoutchouc is obtained from the purest specimens of isoprene and this is true irrespective of the method used for the preparation of the isoprene. Artificial caoutchouc, like the natural product, after drying, must be retained in an atm. of CO<sub>2</sub> and must be protected from light, otherwise it soon becomes brittle. The formula,  $(C_{10}H_{10})x$ , only applies to artificial caoutchouc which has been prepared in an atm. free from O. Specimens formed in contact with air contain O, as also does natural caoutchouc and it is probable that the (b) form mentioned above is "ordinary" caoutchouc which has become coated with the oxidation product. This latter is supposed to be impervious to ordinary, organic, liquid media and, consequently, the unchanged caoutchouc cannot dissolve in them. [Note. For some reason, not stated, the author speaks of the formation of this substance as "autooxidation" and refers to the product as the "autooxidation product." His formulas for isoprene, for its polymerization compd. and for the varieties of pure caoutchouc, show, however, that these substances are free from O. ABSTR.] Comparative vulcanizing expts. (cold process) indicate that probably the best specimens of isoprene caoutchouc are produced when polymerization takes place at the lowest possible temp, and without the addition of any foreign substance. A comparison of the "tetrabromides" of natural and artificial caoutchouc shows that the latter is more readily sol. in CS<sub>2</sub>; other slight differences are also mentioned. In both cases different specimens of the bromides vary in comp.

and it is difficult to prepare from either variety of caoutchoug a bromo derivative of the formula,  $C_{10}H_{16}Br_4$ . The nitrosite (c),  $(C_{10}H_{18}O_7N_2)_2$ , (decomp. 158-62°) from natural zaoutchouc has been described previously. It is also formed from isoprene caoutchouc, but with somewhat greater difficulty. Both varieties of caoutchouc agree in their behavior towards O<sub>2</sub> (12%). The diozonide, C<sub>10</sub>H<sub>16</sub>O<sub>6</sub>, which is formed, when decomp. with hot H<sub>2</sub>O, gives levulic acid (3.2 g.), levulic aldehyde (1.5 g.), resin (0.4 g.), loss (2.2 g.). These yields were from 7 g. of ozonide; they vary somewhat in different expts. There are indications that isoprene caoutchouc contains an isomeric caoutchouc. The distillation of natural caoutchouc (0.1-0.2 mm.) has been described by E. Fischer and C. Harries (Ber., 35, 2162 (1902)). Exactly similar results have been obtained with isoprene caoutchouc. Other substances, besides caoutchouc, are formed by the polymerization of isoprene and several of them have been described by other chemists. The author has isolated from the mixture a kydrocarbon, C<sub>10</sub>H<sub>16</sub>; b<sub>14</sub> 63-5°;  $d_{18}^{18}$  0.8451;  $n_{\rm D}$  1.47408;  $n_{\rm g}$  1.47113;  $n_{\rm r}$  1.48860, all at 18°. It contains but little dipentene. Yield, variable. Ozonide, oily. Butadiene caoutchouc, (C<sub>2</sub>H<sub>12</sub>)x, is prepared from butadiene alone, in a vacuum at 100-10°, or with glacial AcOH, at 110-20°, during 10 dys. in each case. The product resembles closely that from isoprene and similar precautions regarding its oxidation are necessary. At 35°, in presence of Na, butadiene is converted into a new, highly insol. variety of caoutchouc which does not react with HNO<sub>2</sub>, or with O<sub>2</sub>. Butadiene caoutchouc, prepared in presence of glacial AcOH, when treated with HNO, in excess, gives a yellow, amorphous nitrosite, decomp. above 80° (gas evolution). A terpene, C<sub>2</sub>H<sub>12</sub>, is formed together with butadiene caoutchouc;  $b_m$  36°;  $d_{4}^{18}$  0.8523;  $n_D$  1.46768;  $n_a$  1.46423;  $n_r$  1.48812, all at 16°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is yellow, in alc. H<sub>2</sub>SO<sub>4</sub> reddish yellow. Ozonide, white and sparingly sol.; it is rather explosive and when b. with H<sub>2</sub>O forms an aldehyde which gives the pyrrole reaction. Dimethylbutadiene is best polymerized to dimethylbutadiene caoutchouc,  $(C_{12}H_{20})x$ , by heating it without a solvent, at 100°, during about 23 dys. The product is distilled in a vacuum (up to 110°); the residual caoutchouc resembles closely that from isoprene, but is somewhat more readily sol. Yield, 32% of the dimethylbutadiene. Bromide, C12H20Br4, gray and amorphous. It dissolves with difficulty in CS, evolves HBr about 130° and blackens at higher temps. Yield, not quant. Nitrosite, C12H12O7N2, yellow and amorphous, becomes brown 120°, not decomp. 200°. Yield, quant. Diozonide, C12H20O6, is prepared in CCl4, during 9 hrs.; thick, clear liquid from AcOEt + petroleum ether. It gives acetonylacetone when decomp. by b. with glacial AcOH. A second ozonide is formed, together with the preceding compd. and is separated by its sol. in AcOEt + petroleum ether. When decomp, as above it gives a mixture of substances containing acetonylacetone. The preparation of a caoutchouc,  $(C_0H_{12})x$ , by the action of Na on butadiene is described above. The substance will be referred to in this abstr. as "butadiene caoutchouc Na" and its analogs will be designated in a similar manner. After the completion of the polymerization the Na is removed by means of dil. alc.; the product is light yellow, its solubility gradually decreases, it may be vulcanized very readily and the resulting material is superior to any other artificial caoutchouc in elasticity and flexibility. Yield, almost quant. In CHCl<sub>2</sub>, with O<sub>2</sub> (about 6-7%), it gives a diozonide, C<sub>2</sub>H<sub>12</sub>O<sub>6</sub>; white solid, which is also said to be an oil. It is readily sol., but is difficult to purify. With O<sub>2</sub> (12-14%) the product consists chiefly of a monoozonide, probably CaH13O3; it is insol. in CHCl2, AcOEt, or glacial AcOH. The primary product with O<sub>3</sub> of the higher conc. appears to be an ozoneoxide, C<sub>2</sub>H<sub>12</sub>O<sub>4</sub>, which is solid and highly explosive. In Et.O it changes to the monoozonide. Decomp. of these ozonides gives substances which were not identified, except that H<sub>2</sub>O<sub>2</sub> is formed from the diozonide. Nitrosite, yellow, amorphous, insol. and of variable composition. Bromide, white powder from CS<sub>2</sub> + ligroin. It appears to be a mixture of C<sub>2</sub>H<sub>12</sub>Br<sub>2</sub> and C<sub>2</sub>H<sub>12</sub>Br.. Yield, variable. Isoprene caoutchouc Na, C<sub>10</sub>H<sub>16</sub>, is prepared from pure isoprene and Na wire (0.02-5 pts.), at 60°, during 50 hrs. and is purified in the manner described above. Yield, practically quant. It resembles isoprene caoutchouc in general properties, but dissolves more easily when recently prepared and may be vulcanized very readily. In CHCla, with O<sub>3</sub> (12-4%), during 10 hrs., it gives a diozonide, C10H16O6; white solid from AcOEt + petroleum ether, it may be powdered; mol. wt., in glacial AcOH, 250-350. By the use of CCls, in place of CHCls, a mixture of mono- and diozonides is formed; they were not separated completely. When decomp. with H<sub>2</sub>O<sub>2</sub> or glacial AcOH the ozonides give a trace of levulic aldehyde, together with a brown oil; it decomp. when distilled and exhibits the pyrrole test only faintly. With HNO, the caoutchouc forms 2 mitrosites, they are separated by means of acetone. The sol. compd., C10H16OaN2, which is formed in much the larger quantity, is a pale yellow powder, darkens 170°, not m. 260°. The nitrosite insol. in acetone does not appear to have been analyzed. Total yield, 200% of the caoutchouc. Bromide, C10H14Br4, white powder from CS2 + ligroin. It resembles isoprene caoutchouc tetrabromide. Yield, 200% of the caoutchouc. Dimethylbutadisne caoutchouc Na, (C12H20)x, is prepared in a similar manner to isoprene caoutchouc Na. at 60°. during 240-88 hrs.; it is very different from dimethylbutadiene caoutchouc and resembles gutta-percha in appearance. Ozonide, thick oil, sol. in CHCl. Yield, 66% of the caoutchouc. When decomp. it gives H<sub>2</sub>O<sub>2</sub>, a little acetonylacetone and a relatively large quantity of substances which could not be identified. This shows that the dimethylbutadiene caoutchouc Na contains some dimethylbutadiene caoutchouc. Nitrosite, C., H., O.N., from AcOEt + petroleum ether. It dissolves readily in Et.O. Yield, 227% of the caoutchouc. Tetrabromide, C12H20Br4, white powder from CS2 + ligroin, evolves HBr 130°, not m. 200°. Yield, 150% of the caoutchouc. These results especially those obtained by the decomp. of the ozonides, show that the caoutchoucs obtained by the use of Na are different from those prepared by direct polymerization. The paper closes with a discussion of the structure of caoutchouc and a reply to Pickles (C. A., 4, 2936). The author still considers that the basic hydrocarbon of natural caoutchouc is 1,5-dimethylcyclo-1,5-octadiene (II). Tilden was, of J. BISHOP TINGLE. course, the first to obtain artificial caoutchouc.

Pine Oil from Soda Cellulose (HILDING). 22.

Turpentine and Rosin in 1911. 22.

Naphthene Acids (ALBRECHT). 27.

# 27. FATS, FATTY OILS AND SOAPS.

## E. SCHERUBEL.

Naphthene Acids. R. ALBRECHT. Chem. Rev. Fett-Harz-Ind., 18, 152-4; 189-90.

The acid-resins from spent H<sub>2</sub>SO<sub>4</sub> are worked up in Baka and the modern European refineries into fuel or asphalt substitutes by freeing them from the excess of acid by live steam and neutralizing them by spent lye or milk of lime, while the acid, now 40-50° Bé., is conc. in special factories to "black acid" and used in fertilizer works for superphosphates. The spent lye from petroleum refining is worked up for naphthene soaps, used in South Russia as textil soaps or as soap-filler, by salting out and subsequent conc. The spent lye from lubricating oils is treated separately or in connection with above acid-resins by neutralization and is then used as fuel, soap filler, or as cheap lubricant; the latter is much used in Germany and England as ubricant for boring on account of its emulsifying property, and it can be converted into water-sol.

oils by addition of NH, or lye and resin-soaps. It is easy to recover fairly pure soaps from the lyes of petroleum refining but difficult to obtain pure naphthene soaps or acids from the spent lyes of lubricating oils on account of the emulsified, unchanged oil adhering to the soaps. An experimental recovery from the lye of Texas machine oil showed only 15% saponifiable (Spitz and Hönig method) and 85% unchanged oil. By dissolving the soaps in alcohol, a sharp sepn. could be made but these naphthene soaps or acids have not found their way yet into the textil industry, probably because a high acid no. is required by the market, and the high mol. wt. of these soaps diminishes the acid no., though they have the advantage of being odorless. When redistilled the Russian spindle-oil naphthene acids and those from Texas oil vielded light colored distillates of weak but characteristic odor and low I no. (16.9-45.3). Their optical activity was practically as high as that before refining, thus refuting Rakuzin's opinion that the activity of petroleum is caused by naphthene acids. Oils showing a high I no. are more difficult to refine and show a greater loss during refining than the oils whose distillates show a low I no. P. ESCHER.

Blown Rape Oil. D. LOHMANN. Chem. Rev. Fett-Harz-Ind., 18, 184-5.—20 l. of nabo oil, known in Mexico as "aceite de nabo," were "blown" by passing compressed air through it cold and hot and samples were taken at intervals for analysis. 10 hrs. of hot blowing at 160° F. have a greater influence than 80 hrs. of cold blowing at 90° F. The final result of 100 hrs. hot blowing at 160° F. had increased the sp. gr. from 0.914 to 0.964, the viscosity (70° F.) from 375 sec. (Redwood) to 2635 sec., and had decreased the I no. (Hübl) from 99.25 to below 62.3.

P. ESCHER.

Copra Examination. Association of German Oil Mills for the Preservation of Common Interests. Chem. Rev. Fett-Harz-Ind., 18, 195-6.—An investigation of the methods in common for fat detns. in copra has led to the adoption of the following: About 500 g. of copra (disintegrated in a mill of the type shown in catalog 1910, No 1065 Alexanderwerk (Division Remscheid). The milling disk used has a diam. of 180 mm.) is preserved in a glass-stoppered bottle; 10 g. are placed in a cartridge of filter paper and extracted in a Soxhlet, whose syphon should not reach higher than 1 cm. above the top of the cartridge, for 4 hrs., with petrol. ether (b. p. up to 60°) on a water bath whose max. temp. should be 75°. The ether is pressed out of the cartridge by pressing a flattened glass rod on the cotton. The cartridge is dried in the oven for 30 min. when its contents are rubbed up with fine-grained quartz and the mixt. refilled into the cartridge, closed with cotton and again extracted for 2 hrs. The oil is then freed from ether by evapn, and dried in the oven at 105-6° and weighed.

P. ESCHER.

A New Method of Analysis by Miscibility Curves. Its Application to Codliver Oil. R. Louise. J. Pharm. chim., 3, 377-85.—The author applied his method of analysis to the exam. of codliver oil. To obviate errors found due to moisture the oils to be examined were kept at 0° for 24 hrs., then filtered through glass wool and dried in vacuo over H<sub>2</sub>SO<sub>4</sub> for 24 hrs., and the acetone used was diluted with H<sub>2</sub>O so that it would give a miscibility temp. of 7° with the standard petroleum oil used in former detns. To 20 cc. of this acetone different quantities of 3-20 g. of the oil was added and the miscibility temps. plotted in curves. In 15 samples of colorless codliver oil the curves were all similar and the miscibility temps. all above 0°. In the brown oils the curves differed in shape from those given by the lighter colored oils and the miscibility temps. were all below 0°. Pale yellow oils were intermediate in these properties. The values obtained were found to be independent of the I nos. or refractometer readings of the oils.

V. K. Chennut.

Comparcial Analysis of Booswax, Especially East Indian and Chinese Booswax. G. Buchner. Seifensieder Ztg., 38, 878, 902.—See C. A., 5, 3176. E. SCHERUBEL.

Stearinometer (KNORR). 1.

Lubricating Oils. 22.

The Saponins (KOBERT). 17.

Fatty Acids of Codliver Oil (Heiduschka, Rheinberger). 17.

Specific Gravity of Wax (FROMME). 17.

Determination of Glycerol (Ross). 12.

Bleaching Olive Oil. 12.

# 28. SUGAR, STARCH AND GUMS.

### A. HUGH BRYAN.

Raffinose Content and Non-sugar Relation (Organic Non-sugar Over Ash) in Raw Beet Sugar. F. Strohmer. Oesterr.-Ung. Z. Zuckerind., 40, 425-41.—From a number of analyses of raw beet sugars, the author concludes that a non-sugar relation below 1.5 does not always indicate an appreciable raffinose content. This fact is true for the second sugars of normal beets. Raffinose does not occur in appreciable quantities in normal raw beet sugar. In products from the molasses process, the raffinose content is higher the smaller the non-sugar quotient (cf. C. A., 5, 3178).

A. H. BRYAN.

Investigations of Colonial Sugars (Cane Sugar) with Notes Especially Regarding the Use of the Wortman Formula. O. Fallada. Oesterr.-Ung. Z. Zuckerind., 40, 448-58.—In a number of raw sugar samples of different origin, the content of reducing sugars varies considerably. In no case is it true invert sugar (equal quantities of dextrose and levulose). In the most cases dextrose does predominate. The Wortman formula for estg. raffinose and invert sugar, also sucrose, in such cases is not applicable for it assumes an invert sugar polarization value for the reducing sugar.

A. H. BRYAN.

Occurrence of an Apparent Undersaturated Sirup in Beet and Cane Sugar. Theodor Koydl. Oesterr.-Ung. Z. Zuckerind., 40, 459.—The occurrence of an undersatd. sirup in raw sugar, according to the author's method for estg. cryst. content by the wash method (C. A., 2, 1903, 2739, 3165; 3, 497, 2250, 2761, 2880; 4, 687, 1112, 1915, 2389), is often used as an argument against the method. New expts. show that this undersatn. is only apparent. Proceeding not from a supersaturated but from an undersatd. sugar soln., one cannot dissolve as much sugar as is given in Herzfeld's solubility table. The apparent undersatd. sirup explains itself in that the sirup residue, which has started from a slight supersatn. by crystg. out at lower temps. or by imbibing water from moist air at ordinary temps. becomes undersatd. and the satn. figures of Herzfeld's table do not apply.

A. H. Bryan.

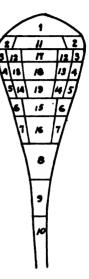
Determination of Reducing Sugars in Cane Sugars for the Refinery or Direct Consumption. C. MULLER. Bull. assoc. chim. sucr. dist., 28, 640–1.—When detg. less than 0.5% of reducing sugar volumetrically, the process may be rendered more sensitive by adding a known amt. of invert sugar, account of which is taken in calcg. the result. Twice the normal sugar-wt. (P) of the sample under exam. is washed into a 100 cc. flask, dissolved, defecated with 2 cc. of N Pb(OAc), soln., and cooled. Twenty cc. of a soln. containing 5 g. of invert sugar per 1. are next added, the liquid mixed, neutralized, made up to the mark, and filtered. The filtrate is titrated against 5 cc. of Fehling's soln. for cane sugars polarizing 98–99° V. The % of reducing sugar is calcd. from the following formula:  $[(n \times 0.005 \times 100)/N - 0.100 \times 100]/2PN$ ; n, the number of cc. of Fehling's soln. used; and N the number of cc. read on the buret.

Results obtained in this way are stated to agree well with those obtained by the gravimetric process (from J. Soc. Chem. Ind., 30, 563).

A. H. BRYAN.

Distribution of Sugar and Non-sugar in the Sugar Beet. A. FLODERER AND A. HERKE. Oesterr.-Ung. Z. Zuckerind., 40, 385-07.—Previous work on this subject is open to criticism on account of poor division of the root for analysis. The authors took large beets uniform in size, both siloed and fresh, and divided into 10 transverse sections. Six of these were again divided into concentric rings as shown in cut. Fifty beets were taken and the resp. pieces of each were mixed together and then analyzed. The highest % of sugar was found in the zones 14, 15, 19, 13, 12, 18, 17, and 16, in order, then decreasing in order in 11, 6, 8, 5, 4, 3, 7, 9, 2, 10 and 1. The total dry substance content does not materially change in any part so the nonsugars increase as the sucrose decreases. In fresh beets the soluble N is less in the interior than in the exterior portion of the beet and ash is in inverse proportion to the sugar content. K and Mg decreased from the top downwards to the region of greatest sugar content and then increased. No definit relation between sugar content and CaO, Na<sub>2</sub>O and P<sub>2</sub>O<sub>4</sub> could be drawn. A. H. BRYAN.

The Bacterial Deterioration of Sugar. Anon. Intern. Sugar J., 13, 414.—A résumé of the work of the different investigators on this subject, with a complete bibliography.



A. GIVEN.

The Correlation between the Sugar and Potash Contents of Cane Juice. H. C. Prinsen Gerrios. Intern. Sugar J, 13, 417; cf. C. A., 5, 389.—The following general conclusions are set forth: Cane juice of high purity produces a small amt. of final molasses and cane juice of low purity produces a large amt. The K contents of molasses from juices of high and low purity do not differ sensibly. The effect of the K content upon the purity of a cane juice is very small (as the amt. of K is very small) compared with the effect of the glucose. The quantity of K in a cane juice remains practically constant after the growth of the cane is completed. The complete disappearance of glucose, which should accompany full maturity, does not take place in cane juices of high K content. "Generally in sugar mills where chiefly a rich and pure cane is crushed, the juice contains little K; while in others, where the juice of even the best ripened canes rizes above a comparatively low figure, large quantities of that element are found."

A. GIVEN.

Preparation of Sugar Lime. F. E. Combs. Louisiana Planter, 47, 101-2.—
Uniform results can be obtained only by slaking the lime in large quantities, making into a thick paste and having a layer of water over it. Burnt lime changes considerably on storing and batches of lime milk made from day to day do not have the same pptg. power.

A. H. BRYAN.

Wholesale Prices of Sugar in Principal Market. Senate Document, 55 (62nd Congress); thru Intern. Sugar J., 13, 435.—A complete statement of the cost of raw and refined sugars for the years 1901 to 1910 for Germany, Austria, France, Russia, Italy, United Kingdom, United States, Cuba, Canada and Java.

A. GIVEN.

Control of Mill Work. J. Lely. Intern. Sugar J., 13, 422.—The author recommends judging mill work by the figure: first mill juice lost per 100 fiber. He shows its application and claims that its use obviates much labor and is more accurate than the methods commonly in use. He gives a table of analyses of different canes and

juices on which the quantity and quality of the mill work were the same, showing the good results of the method.

A. Given.

Experiment with Sodium Chloride Fertilizer for Sugar Beets. F. STROHMER AND O. FALLADA. Oesterr.-Ung. Z. Zucherind., 40, 525-41.—In addition to earlier expts. (C. A., 3, 1601), the authors have tried the substitution for Chile saltpeter of equiv. quantities of N as NH<sub>4</sub>NO, and Na as NaCl. The results show beets of equal quality and tonnage in both cases. Smaller differences are noted in the Na and Cl content of the beet, for with NaCl fertilization somewhat higher were obtained.

A. H. BRYAN.

Experiments with "Shelled" Beet Seed. JOREF DYARFAS. Oesterr.-Ung. Z. Zuckerind., 40, 398-413.—On shelling 2 kinds of beet seeds by a Kuhne machine, a loss in wt. of 25.6 and 38.5% resulted. Germination tests show no particular advantage. The shelled seeds develop more rapidly in field tests but the beets have a greater tendency to "shoot." The tonnage is greater with shelled seed but there is no particular difference in sugar content.

A. H. BRYAN.

Determination of Starch in Spent Grain (KREUZER). 16.
Féry Refractometer. 1.
Resistance of Peroxidases (ASPIT, GAIN). 11.
Determination of Malic Acid (YODER) (DUNBAR, BACON). 12.
Colorimetric Estimation of Sugar (AUTENRIETH, MÜLLER). 11.

## 30. PATENTS.

Abstracts of patents are included under the foregoing divisions of the Journal. The abstracts of U. S. Patents are prepared by Earl T. Ragan, those of the British, French and German Patents by O. D. Swett and those of Canadian Patents by Russel S. Smart, of Fetherstonhaugh & Co., Ottawa (5 Elgin St.). The abstracts of French Patents are prepared, with permission, on the basis of the abstracts in Moniteur scientifique, those of German patents, with permission, on the basis of the abstracts in Chemisches Zentralblatt.

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## CHEMICAL ABSTRACTS

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No. 21

#### 1. APPARATUS.

L. C. JONES.

Improved Soxhlet Condenser. O. SILBERRAD. Chem. News, 104, 54.—This saves trouble in making the cork joint between the top of the extractor and the reflux condenser, by inserting a bulbous Walters condenser into the open end of the Soxhlet tube. A sketch gives the arrangement.

W. H. McLauchlan.

A Simple Check-valve for Water Suction Pumps. R. BEHREND. Organ.-Chem. Lab. Techn. Hochs., Hanover. Chem. Ztg., 35, 807.—A short glass tube of about 10 mm. diam. is drawn to about 5 mm. diam. for half its length. Into the larger end is slipped a tube made tight by means of a piece of rubber tubing extending slightly beyond the end of the inner tube. Between the rubber tubing and the narrow end of the outer tube is the body of the valve, made of a piece of tube drawn to a stem at each end and sealed.

J. H. Moore.

Recent Progress in Calorimetry. W. P. White. Met. Chem. Eng., 9, 202-4, 296-8, 449-52.

J. J. M.

Formation of Nitrous and Nitric Acids from Air. G. D. VAN CLEEFF. Chem. Weekblad., 8, 612.—A lecture app. consisting of a glass globe with tubulures for corks on opposit sides and a long stem on one side midway between the tubulures. The stem passes through a cork in a cylinder, the end dipping into H<sub>2</sub>O; the cork carries a 2nd tube for connecting with pump for aspirating air through the app. The poles of a Ruhmkorff coil, connected with a battery giving 3 amp. current, pass through corks in the tubulures, the spark being at the center of the globe. A glass tube through one tubulure delivers the air just above the spark. After a few minutes the presence of HNO<sub>2</sub> in the H<sub>2</sub>O may be shown with FeSO<sub>4</sub> soln.

J. H. Moore.

High Temperature Measurements. F. T. HAVARD. Mining J., 92, 447-9.—
A resume of methods of measuring high temps with pyrometers and by calorific calculation together with synopses of the principles of operation of the instruments.

ROBERT KANN.

A New System of Gas Lamp Ignition. R. F. Pierce. Illum. Eng. (N. Y.), 6, 312.—The catalytic action of a certain Pt alloy at about 500° is embodied in an igniter used with inverted gas burners. A hollow tube with air vents in the side, and a baffle plate on the end, contains a delicate Pt-alloy filament, connected to the terminals of a dry cell. The tube mounted above the crown of the burner receives small amts. of buoyant gas through the openings around the baffle plate, which on being mixed with air is ignited when it strikes the hot filament. Simultaneously with the opening of the gas valve the circuit of the igniter is completed, being broken when the chain or other valve control is released. Simplicity, low voltage, ease of filament replacement, long life, and a possibility of remote control when combined with magnetic valve attachment, are among its valuable properties.

W. L. VAN KEUREN.

Optical Pyrometry (MENDENHALL). 2.

Drying in Vacuum (VIOLA). 28.

A New Consistometer (CROSBY). 20.

WAGNER, P.: Der Fall Soxhlet. Darmstadt: J. Waitz. 1.80 M.

Ger., 235,541, July 9, 1910. E. Fuld and E. Schesinger, Berlin. In colorimstry, two solns. of different colors, which transmit two or more maxima, clearly sepd. from one another, corresponding to the same spectral elements, are so disposed that the one color is employed in constant thickness and the other in measurably changed thickness, so that the greatest color contrast, resulting from the adjustment of the thickness of the 1 color, is determined.

#### 2. GENERAL AND PHYSICAL CHEMISTRY.

JOHN JOHNSTON.

Walther Victor Spring. F. SWARTS. Chem. Ztg., 35, 949-50.—Obituary. J. J. M.

The Measurement of Infinitesimal Quantities of Matter. W. RAMSAY. J. physique., [5] 1, 429-42.—A lecture delivered before the Soc. fransçaise de physique.

J. C. HOSTETTER.

Chemical Philosophy of the High School Text-books. W. LASH MILLER Science, 34, 257-63.—Address before the Division of Inorg. and Phys. Chem. at the Indianapolis meeting of the Am. Chem. Soc.

J. JOHNSTON.

Rectilinear Diameter for Oxygen. E. Mathias and H. Kamerlingh Onnes. Leiden. Compt. rend., 151, 213-6, 474-5; K. Akad. Wetenschappen, 1910, 1039-57 (Dutch).—After a discussion of the surface representing the state of gases in the neighborhood of the critical temp., the authors describe expts. in which they detd. the ds. of liquid O and of the saturated vapor, with the following results: —210.4°, 1.2746 (liq.), 0.0001 (vapor); —182.0°, 1.1415, 0.0051; —154.51°, 0.9758, 0.0385; —140.2°, 0.8749, 0.0805; —129.9°, 0.7781, 0.1320; —123.3°, 0.6779, 0.2022; —120.4°, 0.6032, 0.2701. The crit. temp. is —118.5°; the crit. d. is calc. to be 0.4292. The results show that the diameter for O is very nearly rectilinear.

J. JOHNSTON.

The Viscosity of Liquids in a State of Turbulent Flow. T. von KARMAN. Physik. Z., 12, 283-4.—A note on the work of E. and M. Bose (C. A., 5, 1545) in which the author demonstrates from theoretical principles that the viscosity in the state of turbulent flow can be calculated from the Poiseuille viscosity constant and the d. if the dependence of time of outflow (or velocity of outflow) on the effective driving pressure is known. Consequently there is no reason for speaking of different viscosities in the two states of flow.

J. Johnston.

Experimental Investigations of the Viscosity of some Organic Liquids in the State of Turbulent Flow. Walter Sorkau. Buenos Aires. Physik. Z., 12, 582-95.— Measurements were made with H<sub>2</sub>O and EtOH at 15° and 25°, and with CHCl<sub>2</sub>, Me<sub>2</sub>CO and trimethylethylene at several temps. below 30°. The results are presented in tables and curves. The last 3 liquids show irregularities at points which appear to vary in a regular way with the temp. The author makes a least square solution for a and b in the equation  $\log z = a + b \log p$ , (z, time of outflow; p, driving pressure) for each liquid in either state at each temp. (cf. E. and M. Bose, C. A., 5, 1545), and presents, and discusses briefly, the results. (See however preceding abstr.)

J. JOHNSTON.

Apparent Association or Molecular Aggregation. J. D. VAN DER WAALS. K. Akad. Wetenschappen, 1910, 78-105, 549-61 (Dutch); Chem. Zentr., 1911, I, 110.—The idea that the mols. are not uniformly distributed in the space occupied by matter

is made use of to account for the abnormalities observed in the case of liquids of large d. in the neighborhood of the critical point. The following modification of the equation as stated is derived:  $p = RT\{1 - (n-1)x/n\}/(v-b) - a\{1-(k)x\}^2/v^3$ . (The total amt. of matter present is 1; x is the fraction present in the form of a mol. complex containing n single mols.; k is less than 1.)

J. JOHNSTON.

The Melting Point of Tantalum. M. VON PIRANI AND A. R. MEYER. Charlottenburg. Ber. physik. Ges., 1911, 540-51.—Great care was used to guard against contamination of the Ta. Measurements were made on lamps, and on strips melted by being made one pole of an arc in vacuo. The temp. was measured by pyrometers, and by extrapolating the temp.-current relation of the lamps. The scale was based on the m. ps. of Au and Pd, for which Holborn and Valentiner's values were taken. The result is given as:  $2850^{\circ} \pm 40^{\circ}$ . With c (Wien constant) = 14600 the calc. temp. would be  $2740^{\circ}$ .

Electrical Heating as a means of Prevention of Superheating of Liquids. J. H. MATHEWS. Lab. Phys. Chem., Univ. Wisconsin. Trans. Am. Electrochem. Soc., 19, 81-90.—The degree of superheating is dependent to some extent upon the nature of the wire,—Pt, Ni-chrome, and "Advance" being better for the purpose than German silver,—but is much more highly dependent upon the condition of the surface. Increase in the no. of bubbles formed by roughness of the surface decreases the tendency to superheat. More perfect circulation and mixing of the liquid produced by an increased rate of b. also decrease this tendency.

C. N. MOORE.

A Comparison of Theoretical and Observed Equilibrium Temperatures in Fog Chambers. E. K. Chapman. Ryerson Physic. Lab., Chicago. *Physic. Rev.*, 32, 561-4.—It seemed probable that the discrepancy previously observed between calc. and actual temps. existing in a fog chamber after expansion was due to the rapid inflow of heat from the metal electrodes. Expts. were made with an app. in which this source of difficulty had been removed. After a slight theoretical correction had been made in J. J. Thomson's equation for computing these temps., the average difference for a series of 18 expts. between calc. and observed temps. was 0.05°.

The Absorption of Hydrocarbon Gases by Non-aqueous Liquids. A. S. McDaniel. Lab. Physic. Chem., Univ. Wis. J. Physic. Chem., 15, 587-610.—The solubility of CH4, C2H4, and C2H4 in a number of org. solvents was detd. with a specially designed app. in which the measurements depended in no way on a knowledge of the v. p. of the solvent. If the solvents be arranged according to their absorptive power the order is the same for all 3 gases. The absorption coeffs. of the same solvents for N, CO, H and CO2, as detd. by G. Just, show different relationships. Hence the sp. nature of the gas may exercize as much influence as the nature of the liquid. When the nature of the gases is such that no "specific action" with the group of liquids is probable, their physical properties may play a greater role than their purely chem. properties. With the hydrocarbons the absorption increases with increasing mol. wt. of homologous liquids, except in the case of the alcs. The temp. coeff. decreases in all cases with increasing temp. In Just's measurements the reverse was the case. This is attributed to the fact that Just did not free his solvents from dissolved air. A bibliography is given.

The Laws of Concentrated Solutions. II. The Estimation of the Degree of Ionization of Electrolytes in Moderately Concentrated Solutions. E. W. WASHBURN. Physic. Chem. Lab., Univ. Illinois. J. Am. Chem. Soc., 33, 1461-78; cf. C. A., 4, 1572.—In calc. the degree of ionization from cond. detns., the neglect of the effect of viscosity may produce errors as high as 8%, even at concs. of o. 1 N and for salts of the simplest

R. H. JESSE.

type. In the present paper the author discusses the application of a correction for viscosity and proposes provisionally as a basis for formulating the correction the relation  $A = k f^m$ , in which A is equiv. cond. of an ion, f the fluidity of the soln., m is a const. not far from unity and dependent chiefly on the nature of the ion, and k is a const. The above relation was found by Johnston to hold is a number of cases in which f was varied by change of temp. (C. A., 3, 2771); it also fits the expts. of Green (C. A., 3, 736) in which f was varied by varying the conc. of sucrose in LiCl soln. Some consequences of the relations are discussed in detail.

J. JOHNSTON.

The Properties of Salt Solutions in Relation to the Ionic Theory. II. Electrical Transference Members. A. A. Noyes and K. G. Falk. Mass. Inst. Tech. J. Am. Chem. Soc., 33, 1436-60; cf. C. A., 4, 3158.—A critical discussion of the methods of detg. the transference number of an ion, followed by a review of previous investigations of transference numbers in solns. of single salts, and of the results obtained. The numerical results are presented in a number of tables; a series of best values is also given.

J. Johnston.

The Conductivity and Ionization of a Penta- and a Hexa-ionic Salt. A. A. Noyes and Robert H. Lombard. Mass. Inst. Tech. J. Am. Chem. Soc., 33, 1423-36.— In this article are presented the results of measurements at 0°, 25° and 50° of the mol. cond. ( $\mu$ ), viscosity ( $\eta$ ) and d. of dil. solns. of the Na4 and Na5 salts of benzene penta-carboxylic acid; also values of  $\mu_0$  obtained by extrapolation, and calc. values of the ratio  $\mu\eta/\mu_0\eta_0$ , which expresses the ionization ( $\gamma$ ), if it be assumed that intermediate ions are not present. The magnitude of  $\gamma$  for the uni-quadrivalent and the uni-quinquivalent salt is in harmony with the principles previously derived from the study of salts of smaller valence products. The cond. of the acid itself at 25° shows that it is a moderately strong acid with respect to both its first and second replaceable H atoms.

J. Johnston.

Dissociation Constant of Oxalic Acid. J. E. ENKLAAR. Utrecht. Chem. Week-blad., 8, 375-82, 443.—The author measured against a normal electrode the e. m. f. at 18° of a H electrode immersed in solns. containing always 0.05 g. oxalic acid per l., to which was added NaOH, in varying amts. corresponding to cones. of 0-33 cc. N NaOH per l. From the values of the H+ conc. derived by this means, the dissociation constant was calc., resulting in values varying only from 0.034 to 0.036, with a mean of 0.035. Hence under the conditions of expt., oxalic acid apparently behaves as a simple binary electrolyte.

J. JOHNSTON.

Conductivity and Viscosity of Aqueous Solutions of Aniline Hydrochloride at 25°. NEVIL VINCENT SIDGWICK AND BERNARD H. WILSDON. Daubeny Lab., Oxford. J. Chem. Soc., 99, 1118-22; Proc., 27, 127.—Measurements are recorded of the d., viscosity and cond. at 25° of solns. of aniline hydrochloride varying in conc. from about 0.1 to 4.1 N. The viscosity of the more conc. solns. is considerable, being for the 4.147 N soln. 3.648 times that of water at the same temp.

J. JOHNSTON.

Solubility of Aniline in Aqueous Solutions of its Hydrochloride. NEVIL V. SIDGWICK, PERCIVAL PICKFORD AND BERNARD H. WILSDON. Daubeny Lab., Oxford. J. Chem. Soc., 99, 1122-32; Proc., 27, 127.—The solubility of aniline in water and in solns. of its hydrochloride of varying strength, at all temps. up to complete miscibility, was detd. The method adopted was the synthetical: mixtures of aniline, water and aniline hydrochloride (or HCl) in known proportions were heated until they became homogeneous and then cooled slowly, the temp. of sepn. being noted. Expts. on the partition of hydrochloride between the two liquid layers at const. temp. showed that the partition coeff. varies greatly (from about 1 to 19 at 25°) with the conc. of the salt. The crit. soln. temp. for aniline and H<sub>3</sub>O is 168°. The full results are presented in tables and diagrams, and discussed; but the discussion cannot be abstracted briefly.

Studies in the Electrochemistry of the Proteins. VII. The Mode of Formation and Ionization of the Compounds of Proteins with Inorganic Acids and Bases. T. BRAILSFORD ROBERTSON. Physiol. Lab., Univ. California. J. Physic. Chem., 15, 521-50.—It is usually conceded that the amphoteric character of the proteins accounts for their behavior towards acids and bases. To the author now "the weight of evidence appears irresistible that some elements in the protein mol. other than terminal NH, or COOH groups are responsible for the acid and base neutralizing power of many proteins." Accordingly it is assumed that the group —CO.NH— (or the corresponding enol form —C(OH) = N—) in proteins is important in relation to their behavior in soln.; this hypothesis is made use of in interpreting the evidence already obtained by the author and others; with the following main conclusions: The salts which proteins form with inorganic acids and bases do not dissociate at the point of union of the inorganic radical with the protein, but elsewhere, within the protein mol. itself; yielding, two or more protein ions, in one or more of which the inorganic radical is bound up in non-dissociable form. On this view the current is transported in both directions by protein ions; this is in harmony with the results obtained from transference expts. with casein in 0.02 or 0.03 N KOH soln., according to which the loss from the cathodal region was about one-half that from the anodal region. From certain cond. detns. with salts of ovomucoid it is concluded that in the formation of salts of proteins with acids (or bases), diamino (or dicarboxylic) radicals are primarily concerned. The ionization of the protein R.NHOCR' is assumed to take place in accordance with equations such as: R.NHOCR' + KOH  $\implies$  RHNOH + KOCR'+; R.NHOCR'+ HCI ≤ RHNCI + HOCR'++. J. JOHNSTON.

The Specific Rotatory Power of Camphor Dissolved in Acetone. H. Malosse. Compt. rend., 153, 56-7.— $[a]_b$  was detd. for solns. of camphor in acetone in which q (g. acetone in 100 g. soln.) varied from 53.44 to 98.66. When q is 53.44,  $[a]_b$  is 50.55; this decreases to a minimum of 48.71 when q is 89.7 and increases again to 50.07 when q is 98.66. Equations for the curve are given in the original.

J. C. HOSTETTER.

Conductivity and Viscosity in Mixed Solvents Containing Glycerol. J. SAM GUY AND HARRY C. JONES. Johns Hopkins Univ. Am. Chem. J., 46, 131-97.-In continuation of the work of Schmidt and Jones (C. A., 3, 2403), the authors have measured the cond. and viscosity at 25°, 35°, and 45° of series of solns. of the chlorides, bromides and nitrates of K, Na, NH, Ba, Sr and Ca in mixtures of water, MeOH or EtOH with glycerol containing 25, 50 or 75% glycerol; also in pure glycerol at the additional temps. 55°, 65° and 75°. The results are presented in about 150 tables and several diagrams. The conds in the mixed solvents are in each case lower than those calc. by the simple mixture law. The cond. and fluidity curves are strikingly analogous at all the temps, and for all the solns, studied. Salts which have the highest power of solvation show the greatest temp. coeffs., and these are greater in the more dil. solns. The mol. conds. of ternary electrolytes in glycerol at low dilns. are usually smaller than those of binary electrolytes under the same conditions, while at high dilutions the reverse is generally true. The majority of the salts studied diminish the fluidity of giveerol, yet certain salts of Rb and NH, increase its fluidity. This is analogous to the behavior of certain salts of Rb, K and NH, in aq. soln. and is probably correlated with the large at. vol. of the cation in these cases. J. JOHNSTON.

Temperature Coefficient of Concentration Cells, in which the Same Salt is Dissolved in Two Different Solvents. A. P. Laurie. Proc. Roy. Soc. Edinburgh, 31, 375–96; cf. C. A., 3, 506.—After an historical introduction, the theory of these cells is developed on the basis of the Nernst and Helmholtz equations. The e. m. fs. of cells containing KI and I<sub>2</sub> in H<sub>2</sub>O at one electrode (Pt) and in EtOH (or  $C_6H_6NO_2$ ) at the other were measured at two temps. but the results cannot be compared with the theory quantitatively

owing to lack of knowledge of the partition coeffs. of the solutes between the solvents and of the latent heats of soln. Using Ag-AgI electrodes and 0.001 N KI in EtOH around one electrode, 0.02 N KI in H<sub>2</sub>O around the other electrode gives zero e. m. f., whereas weaker aq. solns. give a current which transfers KI from alc. to water and stronger aq. solns. vice versa. If both alc. and water are saturated with KI, there is an e. m. f. which shows that the electrolytic transfer of the ions across the liquid junction must be accompanied by mixing of the solvent, which is the only possible source of energy in this case. This shows that the ions must be combined with alc. or water or both. A 2 N KI soln. in H<sub>2</sub>O gives zero e. m. f. against satd. soln. of KI in EtOH.

GRINNELL JONES.

Value of the Components of the Electromotive Forces of Voltaic Cells. G. Guglielmo. Atti accad. Lincei, 19, II, 572-7; Chem. Zentr., 1911, I, 860.—Abs. potentials can be represented by the equations V' = C'/e + K, V'' = C''/e + K, where C', C'' are the heats of combination, expressed in joules, of each metal with the electronegative component of the soln., and e the amt. of electricity the passage of which corresponds to the chem. effect of the heats of combination. K can be detd. by means of the equations K = dV''/TdT = dV''/TdT or K = c'/e = c''/e, c', c'' in the last 2 being the local heat effects at the 2 electrodes corresponding to the passage of the current e (Peltier effect). Using a few results obtained by himself, but depending chiefly on data found in the literature, the author finds K approx. 0.2 v. This energy he considers as the work done in sepg. (in positive or negative sense) the metal and its elec. charge, while the first term in the expressions for V', V'' corresponds to the heat of combination of metal and anion. Chas. A Rouiller.

Vapor Pressures in Binary Systems with Partial Miscibility of the Liquids. P. KOHNSTAMM AND J. TIMMERMANS. Proc. K. Akad. Wetenschappen, 1911, 865-81 (English).—The authors test some conclusions which van der Waals (C. A., 3, 137; Arch. neerland sci. exact nat., 13, 249) reached by mathematical analysis. They have measured the vapor pressure at several temps, of the following binary mixts, with 2 liquid phases up to the temp. of complete miscibility (critical end point) given: Cyclohexane + C<sub>0</sub>H<sub>4</sub>NH<sub>2</sub>, 31.09°; C<sub>0</sub>H<sub>14</sub> + C<sub>0</sub>H<sub>5</sub>NO<sub>2</sub>, 20.40°; and also the b. p. of many single phase mixts. of the following liquids:  $C_0H_{14} + C_0H_0NH_2$ , (crit. end point 68.9°);  $(CH_2)_6 + C_8H_3NH_2$ , iso- $C_3H_{12} + C_8H_8NO_2$ , 32.1°. They discuss the shape of the p, T projection of the 3-phase curve with 2 liquid phases and the connection between the shape of the plait-point-line and the existence of a max. in p, x section of the surface of satn. The expectation pronounced by van der Waals that the line of the 3-phase press. would touch the plait-point-line in the crit. end point is in contradiction with expt.; further theoretical consideration proves this expectation to be unfounded, and results in 2 other rules which are both confirmed by expt. In general, the rule of van der Waals that splitting up of a plait-point-line only occurs in systems from the middle region of the diagram of isobars, is corroborated by expt., but other cases occur. In opposition to the theories of Ostwald, Speyers, Dolezalek, and others, but in agreement with van der Waals, mixts. of normal substances can possess p, x lines with a point of inflection, and even with more than one, when we approach the region of non-GRINNELL JONES. miscibility.

Fusibility Curves of Gaseous Mixtures: Systems Formed by Carbon Dioxide and Hydrogen Sulfide with Methyl Alcohol and Methyl Ether. G. BAUME AND F. L. PERROT. Compt. rend., 152, 1763-5.—A study of the systems Me<sub>2</sub>O-CO<sub>2</sub>; MeOH-CO<sub>2</sub>; MeOH-H<sub>2</sub>S; and Me<sub>2</sub>O-H<sub>2</sub>S indicated no compounds in the first 3 systems; but the curve of the last system showed a well-defined maximum at —148.5°, corresponding to a new oxonium compound, Me<sub>2</sub>O,H<sub>2</sub>S.

R. H. JESSE.

Diffusion of Electrolytes in Colloids. LUIGI ROLLA. Ist. chim. gen. univ. Genova.

Atti accad. Lincei, 20, II, 47-51.—In a cylinder of pure gelatin soln. (6%) in a glass tube open at both ends were placed 2 Ag electrodes, uniformly covered with a layer of AgCl and provided with numerous small holes, parallel to the base of the gelatin and sepd. from each other by a distance 2a, the lower one being at a distance X above the base of the gelatin. If now, in the plane of the base of the gelatin an electrolyte is circulated at const. conc. K for a time  $\theta$ , and then pure  $H_2O$  is immediately substituted for the electrolyte so that the conc. at the base of the gelatin is maintained at zero, and if the coordinates giving the position of the 2 electrodes be represented by S + a and S - a, resp., and the instant at which the max. e. m. f. is manifested by  $\theta$ , then the diffusion coeff. in Fourier's equation  $(\partial c/\partial t - k^2 \partial^2 c/\partial t^2)$  becomes  $k^2 - k^2 \partial^2 c/\partial t^2$  $a \, B^2/2(a + B)\theta$ . Expts. with 1-2.5 N KCl and NaCl as electrolytes at 4-12°, Ebeing 1.25 cm., 20 0.5 cm. amd  $\theta$  0.4-0.7 hr., gave, after reducing the values obtained to 18° on the assumption that the mobility of the ions and the osmotic pressure of the dissolved substances have the same temp. coeff. in gelatin as in H<sub>2</sub>O, 1.33 and 1.01 for k2 (cm.2/day) for KCl and NaCl, resp., in good agreement with Oholm's results (Z. physik. Chem., 50, 308). CHAS. A. ROUILLER.

Mechanism of Electrolytic Transfer. J. W. RICHARDS. Trans. Am. Electrochem. Soc., 19, 119.—According to the author's theory, the passage of an elec. current either through an electrolyte or through a metal is the same phenomenon. Decomp. of the electrolyte occurs at the electrodes; the differences in conc. resulting are instantaneously equalized by solubility and diffusion.

G. R. FONDA.

The Relation of Surface Action to Electrochemistry. H. E. PATTEN. Trans. Am. Electrochem. Soc., 19, 359-80.—After a discussion of the general equation for surface energy, the author considers the electrochem. aspects of surface action in respect to the migration, settling and resuspension of suspended particles. G. R. F.

Influence of the Acidity of Aqueous Solutions on the Surface Tension of the System Water-Oil. W. Reinders. Delft. van Bemmelen Gedenkboek, 333-41.—The author has studied by the drop-wt. method the surface tension at 18° of oil against aq. solns. of H<sub>2</sub>PO<sub>4</sub> to which varying amts. of NaOH were added, so that while the PO<sub>4</sub> conc. was in each case o.1 N, the Na<sup>+</sup> conc. varied from o-o.1 N. It was found that the variation of drop wt. is parallel to that of log [H<sup>+</sup>], although the relation between these quantities is not linear over the whole range of concs. of H<sup>+</sup>. This regularity was observed with various oils. However, with oils containing oleic or stearic acids (the latter especially) the drop wt. is smaller; with the higher acid concs. of the aq. phase, such oils flow out in a stream, without forming drops at all. Truly stable suspensions are obtained only when the surface tension is very small.

J. J.

The Analogy between Swelling (Imbibition) and Mixing. I. J. R. KATZ. Proc. K. Akad. Wetenschappen, 1911, 958-75 (English).—On the view that the processes of swelling and of mixing are essentially the same, the author has made measurements of the dependence of heat of imbibition, vapor tension and vol. contraction on the degree of imbibition i (the no. of g. H<sub>2</sub>O taken up by 1 g. of the swelling substance); and also analogous expts. with miscible liquids. The systems investigated were, on the one hand, H<sub>2</sub>O with cellulose, casein, woody fibers, gelatin, etc.; on the other hand, H<sub>2</sub>O with H<sub>2</sub>SO<sub>4</sub>, H<sub>2</sub>PO<sub>4</sub> and glycerol. In each case the curves obtained with the swelling substances are remarkably similar to those found with the mixtures. These similarities lead the author to the conclusion that amorphous swelling substances differ from liquids only in viscosity. II. Swelling Crystals and Mix-crystals. Ibid., 975-81.—The author has detd. the variation of vapor pressure with i for the substances carboxyhemoglobin, a globulin from seeds of Cucurbita Pepo, Bence-Jones' albumose, and amylodextrin; these are cryst substances which show the phenomena of swelling. In each case the curves obtained are S-shaped, continuous,

similar to each other and to the analogous curves obtained w...h amorphous swelling substances. Analogous detns. were made with 4 substances which are known to form solid solns. with water; namely quercitrin, Ca chabasite, Th oxalate, and basic Zr oxalate; the curves obtained resemble closely those obtained with the swelling substances. These expts. show that there is no sharp line of distinction between swelling crystals and mix-crystals, and confirm the hypothesis that swelling is due to the formation of a solid soln. of water in the imbibing substance.

J. J.

A New Method for Proving the Validity of the Boyle-Gay Lussac Law for Colloidal Solutions. II. The Svedberg and Katsuji Inouve. Chem. Lab., Univ. Upsala. Z. physik. Chem., 77, 145-91.—In this article a full description is given of extended series of expts., similar to those previously described (C. A., 4, 2596), with hydrosols of Au of various cones. The main conclusions follow: The compressibility  $(\beta/\beta_0)$  decreases with increasing cone. and with increasing size of particle. At great dilns.  $\beta/\beta_0$  is unity, in accordance with the theory; in other words, the gas laws hold at great dilns. The effect of added foreign material (HCl, NaOH, urea, sugar, gelatin at various small cones.) on the compressibility is inappreciable. Consequently, (1) the compressibility is independent of the viscosity of the dispersive medium, and (2) Dalton's law of the independence of partial pressures is valid for colloidal solns. Similar expts. with gum gamboge hydrosols yielded conclusions similar in every respect to the above, except that at the greater cones, the deviations from the theoretical values are in this case much smaller.

Concentric Rings in Filter Paper. E. Lenk and H. Brach. Vienna. Z. Chem. Ind. Kolloide, 8, 325-6.—The formation of concentric rings of a ppt. when 2 salts react in gelatin is a familiar phenomenon (H. Schade, Ibid., 4, 175 (1909); Hatschek, C. A., 5, 2458; R. E. Liesegang, C. A., 5, 404, 1703). The author has devized a means of obtaining the same effect on blotting paper by fixing a dropping funnel above a piece of blotting paper which has been soaked in a soln. of 1 of the salts, e. g., K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, then dropping onto the paper a soln. of the other salt, AgNO<sub>2</sub>. A ppt. of Ag<sub>2</sub>CrO<sub>4</sub> in the form of a circle first appears and outside that circle is a ring free from K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, due to the diffusion of the K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> and its removal by pptn. A 2nd drop of AgNO<sub>2</sub> diffuses beyond the ring and forms a larger concentric ring of Ag<sub>2</sub>CrO<sub>4</sub>. Thus a series of concentric rings of Ag<sub>2</sub>CrO<sub>4</sub> sepd. by rings free from a ppt. or either of the salt solns. is formed.

The Preparation of Colloidal Vanadic Acid by a New Dispersion Method. E. MÜLLER. Stuttgart. Z. Chem. Ind. Kolloide, 8, 302-3.—If molten vanadic acid is suddenly poured into H<sub>2</sub>O a brown colloidal soln. of vanadic acid results, which is reversibly pptd. by NH<sub>4</sub>Cl. On evapn. of the soln. the vanadic acid seps. on the walls of the flask as a thin skin, insol. in water, but much more chemically active than the usual form. The theory that the peculiar solubility effect is due to the sudden chilling of the molten mass is confirmed by the fact that when molten vanadic acid is suddenly cooled by immersing the crucible containing it in H<sub>2</sub>O, the solid then gives the same colloidal soln. An effort to prepare a colloidal soln. of AgCl by the same method has not been successful.

Adsorption of Arsenic by the Hydroxides of Aluminium and Iron. G. LOCKEMANN AND M. PAUCKE. Leipzig and Berlin. Z. Chem. Ind. Kolloide, 8, 273-88.—Biltz and Behre (Ber., 37, 3138 (1904)) have shown that the reaction between arsenious acid and freshly pptd. Fe hydroxide is an adsorption phenomenon of the type  $y^{\pm}/x = c$  where y is the quantity of arsenious acid adsorbed, x the quantity remaining in soln. The most complete adsorption of arsenious acid by Fe hydroxide occurs at low temp. and when the Fe has been pptd. by stoichiometric quantities of NH<sub>4</sub>OH. An excess of NH<sub>4</sub>OH or pptn. by KOH or NaOH decreases the adsorption. The arsenious acid

is completely adsorbed from solns. containing up to 10 mg. As per 100 cc.; for quantities greater than this the equation  $E = \beta A^{\beta}$  expresses the conditions necessary for complete adsorption, where E = mg. Fe(OH)<sub>2</sub> and A = mg. As in 100 cc.,  $\beta = 62$  at  $0^{\circ}$ , 80 at  $25^{\circ}$ , and 120 at  $80^{\circ}$ ;  $\beta = 0.6$  at all temps.

Ultramicroscopic Investigation of some Colloid Coagulations by Electrolytes. G. Wiegner. Z. Chem. Ind. Kolloide, 8, 227-32.—The process of coagulation of a colloid by electrolytes was watched under the ultramicroscope by counting the number of ultramicroscopic particles per cc. after the addition of increasing amts. of electrolyte. In the case of cow's milk and colloidal Au, using H<sub>2</sub>SO<sub>4</sub> or BaCl, as coagulant, the no. of particles increases slightly at the beginning of coagulation, then remains constant up to a certain electrolyte conc. beyond which it decreases rapidly. The process thus consists in the addition of the smallest amicron to the larger submicrons acting as seeds. The mass of the submicrons is thus increased without changing the no. until finally all the amicrons have disappeared, when the no. of submicrons begins to decrease.

The Action of Salts in Heterogeneous Systems. S. B. Schryver. London. Z. Chem. Ind. Kolloide, 8, 233-8.—If the solubility of globulin in salt solns is due to adsorption of salt by the products of hydrolytic cleavage of the globulin, thus preventing a reunion of the products of hydrolysis, then theoretically those salts which in soln. cause the largest decrease of the surface tension of H<sub>2</sub>O (a phenomenon of adsorption) should be the best solvents for globulin. It was found by expt. that the efficiency of salt solns, as solvents for the serum globulin of the horse was greatest for those salts giving the lowest surface tension when dissolved in H<sub>2</sub>O, provided only inorg. or org. salts were being compared. Of an inorg. and an org. salt soln. having the same surface tension, the former was much the better solvent because of its greater rate of diffusion. In like manner salts interfere with the pptn. of Witte's peptone by CH<sub>2</sub>O, the salt of lowest surface tension having the most marked effect, due to an adsorption of the salt by the peptone, thus preventing aggregation of the organic mols. The critical soln. temp. of different salts and phenol decreases as the surface tension of the salt soln. decreases. The solubility of leucine and phenylalanine in salt solns. was found to vary inversely with the surface tension of the salt soln. H. ISHAM.

Action of Crystalloids on Colloidal Sulfur. SVEN ODEN. Chem. Lab., Univ. Archiv. Kemi. Min. Geol., 3, No. 31, 19 pp. (Swedish); Chem. Zentr., 1910, Upsala. II, 1022; cf. Svedberg, C. A., 3, 1612.—The S concs. which are in equil. with NaCl solns, of various concs. vary with the previous history of the colloidal S soln.; in order to obtain comparable results it was therefore necessary to work with a single S soln. If s is the percentage amt. of S in soln., n the conc. of NaCl in centimols. per 1., the formula  $sn^{2.8} = 32810$  holds for values of s between 5.4 and 0.07% and for cones. of NaCl between 1.2 and 4.3%. The influence of various cations upon the coagulation is great; that of anions is small. The author denotes by the term critical salt conc. that conc. at which the milkiness caused by the pptd. S will not disappear. This conc. can be detd. sharply; its value for a number of alkali salts is given. The S pptd. by salts of Na and NH<sub>4</sub> is easily sol. in cold water; but all ppts. of S are insol. in hot water. The higher the at. wt. of the cation, the greater its coagulating power. Anions, on the other hand, increase the dispersion. The action of bases resembles that of salts; dil. alc. does not coagulate, conc. alc. does. The speed of coagulation of S solns. of different conc. is very different. A soln. which has become milky may be cleared by the addition of acid. Investigation of the crit. salt conc. (s) of KCl in presence of a number of mineral acids showed that the relation between them is given by s - $Am^{\beta}$ , where m is the mol. conc. of the acid, and A and  $\beta$  consts. With formic acid, however, s increases linearly with m up to concs. of 8 N. J. JOHNSTON.

Radiations which Decompose Water and the Extreme Ultraviolet Spectrum of the Mercury Arc. A. Tian. Compt. rend., 152, 1483-5; cf. C. A., 5, 813, 2207.—Spectroscopic measurement of the Hg arc reveal a large triplet 2000, 1971, and 1941  $\mu\mu$  and the lines 1846, 1848 and 1851. From the author's expts., it is the last three which decompose H<sub>2</sub>O into H<sub>2</sub> and H<sub>2</sub>O<sub>2</sub>. The Al spark spectrum which has the lines 1854, 1857 and 1862 also effects this decomp.

R. H. JESSE.

Quick Quenching Spark Gaps for Close Coupling. Hans Boas. Ber. physik. Ges., 1911, 527-39.—Hg gaps for impulse excitation are defective on account of the increase of the vacuum with time; the diminution of damping which results has generally been laid to other causes. Spark gaps of very short length (0.1 mm.) between metal plates are generally used, and for best working require a rather definit degree of coupling between the primary and secondary circuits, in practice not much over 19%. Shorter gaps can not well be used with the soft metals, Ag and Cu, generally used in order that their high thermal cond. may exert a cooling action and so help the quenching of the spark. Boas doubts the practical importance of this cooling action; with gaps of Pt-Ir he can use a shorter distance, and get 40% coupling.

W. P. WHITE.

Notes on Optical Pyrometry. C. E. MENDENHALL. Univ. Wisconsin. *Physic. Rev.*, 33, 74-6.—A description of (1) a rotating sectored disk which can be conveniently mounted on a Holborn pyrometer, thus avoiding the difficulties and uncertainties which come when different absorbing glasses are used. (2) A test of a method of calibrating the pyrometer by means of this disk and *one* temp., using Wien's law. The uncertainty here was whether this law could be applied with the red glass, whose transmission is not strictly monochromatic. A comparison calibration, using a spectroscopic eye-piece in place of the red glass, gave results agreeing to 2°, the limit of accuracy, whence it appears that the simple method is valid. (3) The eye-piece with which the previous test was made. It is similar in principle to one independently introduced by Henning, but is simpler.

W. P. W.

A Study of the Structure of Spectral Rays with the Aid of a Large Dispersion Appearatus. G. MESLIN. Ann. chim. phys., 24, 87-133.

J. J. M.

Annual Review of Optics. L. HOULLEVIGUE. Rev. gen. sci., 22, 650-5. J. J. M.

Measurements of the Zeeman Effect. Magnetic Displacements of the Lines Emitted by Substances in the Gaseous State. A. Corron. Le Radium, 8, 33-43.—A review.

Merce Randall.

Investigation of the Magnetization of Liquid and Solid Oxygen. H. KAMERLINGS ONNES AND A. PERRIER. K. Akad. Wetenschappen, 1910, 799-835.—Fleming and Dewar concluded from their measurements that the susceptibility (X) of liquid O at its b. p. fitted in with Curie's law (deduced from expts. with gases at temps. between 20° and 450°) according to which X is proportional to T (the abs. temp.). The present measurements show however, that for liquid O (between  $-183^{\circ}$  and  $-208^{\circ}$ ) the relation  $X = 0.002284/\sqrt{T}$  holds; and for solid O (from  $-259^{\circ}$  to the m. p.)  $X = 0.001690/\sqrt{T}$ . Hence, at the f. p., the susceptibility of the liquid is 1.3 times as great as that of the solid. It appears that the above relation for the liquid goes gradually over into the Curie law between  $-183^{\circ}$  and 0°.

J. JOHNSTON.

Determining the Density of Solids (MERWIN). 8.

Periodides and Perbromides (HERZ, BULLA). 6.

Combining Power of Chlorides (HERRMANN). 6.

Temperature Coefficients of Resistance (SOMERVILLE). 4.

Surface Tension of Molten Glasses (TILLOTSON). 19.

Diffusibility of Cholesterol Esters (BOAS, ROSENBLOOM). II.

Density of Liquid Sugar (SCHWERS). 28.

Absorption Spectra of Cinchonine (DOBBIE, LAUDER). 10.

Absorptive Power (CRYMBLE, et al.). 10.

Conductivity of Malonic Acids (MELDRUM). 10.

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## 3. RADIOACTIVITY.

#### UPPMAN SCULLING

Radioactivity. A. Debierne. J. Am. Chem. Soc., 33, 1388-1402.—A summary of the important discoveries, relationships, and theories of radioactivity.

H. SCHLUNDT.

Radioactivity of the Sediments from the Teplitz-Schönau Springs. J. STERBA. Chem. phar. Inst., Univ. Prague (Bohm). Jahrb. Radioakt. Elektronik., 8, 23-34.—Sediments and incrustations from the important springs of this group were examined for radioactive constituents by an electroscopic method as well as chemically. The deposits from the different springs were all found to contain radioactive elements. The activity of the deposits varied (approx.) inversely as the activity of the H<sub>2</sub>O sam-

ples. The emanation, as shown by the curves of decay of the active deposits obtained upon the walls of the ionization chamber, consists of a mixt. of Ra Em. and Th Em. for some of the springs, of pure Th Em. in a few cases, and of Ra Em. alone in others. The expts. dealing with the chem. sepn. of the active constituents from relatively large quantities (50 kg.) of the sediments failed to show the presence of U in any of them. Ra, Th, radio-Th, and radioactive Pb and Bi were isolated.

H. SCHLUNDT.

Chemical Reactions of the Radioactive Elements. B. SZILARD. Lab. of Mme. Curie, Fac. Sci., Paris. Radium, 7, 366-72; Magyar Chem. Lapja, 2, 3-5.—Reactions in which a radioactive element is carried down by the pptn. of a salt in soln. are ionic. The grains of the ppt. once formed produce 1 of the ions; the other ion is produced out of the electrolytic ion of the radioactive element. These particles or ions seem to have different properties from those of the elements. The conditions of a reaction once established, it serves to identify the radioactive element in the same manner as ordinary reactions serve to identify the elements.

MERLE RANDALL.

Scattering of Alpha and Beta Particles of Matter and the Structure of the Atom. E. RUTHERFORD. Phil. Mag., 21, 669-89.—A very thin layer of metal produces a much larger scattering of both  $\alpha$  and  $\beta$  rays (greater for  $\beta$  than for  $\alpha$  particles) than a multitude of small scatterings, due to the successive encounters with numerous ats. would indicate. For a layer of Au foil only 0.0004 cm. thick the probable angle of deflection for the  $\alpha$  particles is less than 1° on the theory of successive encounters, whereas an appreciable no. of particles are deflected 90°. It is supposed then that these deflections are produced by the particles passing through the atoms where they are subject to powerful elec. fields. The author assumes for the present an atom consisting of a positive charge conc. at a point and surrounding it a group of negative corpuscles. He calculates the probability of a single deflection through any angle, and enters upon the case of compound scattering and the alteration of velocity in an at. encounter. The results of Geiger and Marsden on "diffuse reflection of  $\alpha$  particles," and "variation of diffuse reflection with at. wt.," and those of Crowther on "scattering of  $\beta$  rays of different velocities" agree qualitatively with the author's theory. This article serves to point out a general line of experimentation for securing quant, data with which to test the validity of the assumptions made on the structure of the at.

ROBT. F. EARHART.

Delta Rays. NORMAN CAMPBELL. Phil. Mag., 22, 276-302.—The slow-moving electrons emitted by a plate covered with a substance emitting  $\alpha$  rays are called  $\delta$  rays. These are believed to be a scondary radiation excited by  $\alpha$  rays in any solid body upon which they fall. The value of  $e/\dot{m}$  was assumed and the measurements on velocity of the  $\delta$  rays studied by the action of an elec. field. The conclusions are: (1) There is no evidence that the speed of the  $\delta$  rays is dependent on the speed of the  $\alpha$  rays exciting them nor (2) of any difference in speed between the emergence and incidence radiations. (3) It is doubtful whether the speed of the  $\delta$  rays varies with the material from which they are emitted. Such a variation seems to have been detected, but considerations of the possible effect of the reflect ion of the  $\delta$  rays at the electrodes renders the prima facie evidence unreliable. (4) There is no evidence that the no. of  $\delta$  rays emitted depends on the material from which they are emitted.

R. F. E.

Measurement of the Range of the Alpha Particles of Uranium by the Method of Scintillations. A. Foch. Lab. of Mme. Curie, Fac. Sci., Paris. Radium, 8,  $101^{-4}$ .—A spherical glass balloon is coated on the inside with a thin film of the radioactive substance, the range of whose  $\alpha$ -particles is desired. The flask is so arranged that it may be evacuated and a gas at any pressure admitted. Exactly in the center of the flask was placed a button of ZnS and outside the flask a telescope through which the

number of scintillations could be observed. If the pressure of the gas within the flask be gradually increased the no. of scintillations will gradually decrease either to a const. value or to zero. With Ra F and a balloon of 78 mm. radius, a minimum was reached at a pressure of 36 cm. Hg in air, which gives a range of 3.8 cm. at atm. pressure. With a balloon of 48 mm. radius and a film of U acetate, the no. of scintillations was zero at 42 cm. The range of the  $\alpha$  particle of U in air at atms. pressure is then 2.68 cm.

Researches on the Cathode Rays. J. MALASSEZ. Ann. chim. phys., 23, 231-75, 397-424, 491-521.—Kaufmann and Simon in ascertaining the value of e/m for cathode rays have each assumed the formula of Schuster which accounts for the kinetic energy of the corpuscles as derived from the fall of potential between the cathode and anode. The author's work consists of: (1) Verification of the exactness of Schuster's assumption; (2) detn. of e/m by the method of Simon; and (3) comparison of the value of mv<sup>2</sup>/e as deduced from the equations of Schuster with the value calc. from the electrostatic deflection. In the introduction, the classical methods employed by different investigators in detg. the value of e/m are reviewed, including the more recent ones of Bestelmeyer, (Ann. Physik, 32, 429 (1907), Classen, C. A., 3, 396 and Wolz, C. A., 4, 273). To ascertain whether the kinetic energy of the cathode particles is proportional to the fall of potential, the author supplements the p. d. in the discharge tube by a known p. d. and measures the deflection of the rays with and without the superimposed p. d. He thus obtains data that verify the assumption of Schuster. The value for e/m obtained by the method of Simon was found to be less than the values found by other methods, namely 1.77 × 107. The reasons for the divergencies are discussed.

Report on the Separation of Ionium and Actinium from Certain Residues and on the Production of Helium by Ionium. B. B. Boltwood. Proc. Roy. Soc. London, (A) 85, 77-81.—The "actionium residues" consisted of 21.2 kg. of wet paste sepd. from 500 kg. of pitchblende residues which had been treated for the removal of Ra. This paste was a heterogeneous mixt. containing considerable PbCl, and gelatinous SiO. and small quantities of Cu, Zn, Fe, Sn, etc. The preliminary treatment of this paste (under direction of Rutherford and Greenwood) was as follows: Treated twice with HCl to remove Pb. HS is used to remove sulfides; the filtrate boiled with HNO, and excess of NH, added and boiled to ppt. hydroxides. Evapn. of the filtrate leaves considerable residue of NH<sub>4</sub> salts (Res. B). The hydroxides were dissolved in dil. HCl and oxalic acid added, with NH, to neutralize. The pptd. oxalates (after 17 hrs.) were ignited at low red heat and the oxides digested with HCl, and a slight insol. residue filtered out. The dild. filtrate was nearly neutralized with NH, and H,O, added. This dried residue (160 g.) was the starting point of the author. Its activity was about 20% of that of the original substance. It contained Ca, rare earths and F (from the H<sub>2</sub>O<sub>2</sub> used). The ppt. was repeatedly treated with H<sub>2</sub>SO<sub>4</sub>, a soln. containing rare earths, an inactive residue of CaSO4 and SiO2 being thus obtained. The soln. was boiled, excess of (NH<sub>4</sub>)<sub>3</sub>C<sub>2</sub>O<sub>4</sub> added, the ppt. (after 24 hrs.) ignited to oxide; weight 0.5 g. The oxides were dissolved in HCl and the rare earths pptd. as hydroxide with excess of NH<sub>2</sub>. The ppt. is dissolved in HCl, soln. dild., boiled and an excess of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> added. After b., the ppt. was filtered out and the treatment with HCl and Na<sub>2</sub>S<sub>2</sub>O<sub>2</sub> repeated 4 times. The final ppt. dried and ignited, consisted of 1.8 g. of highly radioactive ThO2. The ionium which it contained was about 3000 times as active as an equal wt. of U oxide. The total av. number of  $\alpha$  particles emitted (as counted by Geiger) by the 1.8 g. was 18.1 × 10<sup>7</sup> per second. Since Rutherford and Geiger have shown the  $\alpha$  particles emitted by 1 g. of Ra to be 3.4  $\times$  10<sup>16</sup> per sec., the amt. of ionium present with the Th, was equal to the amt. in equil. with 5.3 mg.

of Ra. An exam. of B (above) showed actinium. The 18 kg. of B were subjected to fractional crystn. to remove the greater part of the salts and the final active residue boiled with HCl and HNO<sub>2</sub> to remove the NH<sub>4</sub> salts. After removing excess acid, the residue, chiefly Ca salts was dissolved in water. Treatment with pure NH<sub>3</sub> gave a ppt. weighing 10 g. after ignition; it was only slightly radioactive at first, but the increase of activity corresponded to that of an actinium prep. from which radioactinium and Act. X had been removed. After 4 months the activity was 20,000 times that of an equal wt. of U oxide, and large amts. Act. Em. were given off. The amt. of actinium was estd. roughly to be equal to the amt. in equil. with 30 mg. of Ra. 1.5 g. of ThO<sub>2</sub> containing the ionium, when sealed in glass tube with pure O<sub>2</sub> and after 125 days heated to bright redness, gave 0.031 cu. mm. of He. 0.0595 cu. mm. He was calc. but the sepn. of He from the ThO<sub>2</sub> probably was not complete.

C. H. V.

Processes in the Transport of Active Precipitates. E. M. WHLLISH. Ber. physik. Ges., 1911, 159-71.—The author interprets the work of Russ (Ber. physik., 1909, 397 (1909)), Kennedy (Phil. Mag., [6] 18, 744), Rutherford (Phil. Mag., [5] 49, 161 (1905)), Franck and Schmidt (Physik. Z., 10, 184 (1908)) and Makower (Phil. Mag., [6] 10, 526) on the passage of radioactive "rest atoms" through gases, under varying conditions of pressure and elec. field on the basis of the following hypothesis: It is assumed that the rest atoms are uncharged, except when associated with a gas ion, and that a gas ion in motion through a gas will be associated for a certain fraction of its existence with various rest atoms, during which time the rest atoms will move in the direction in which the ion is moving. The fact that the active deposit in an elec. field is found almost entirely on the cathode must be explained by assuming that the particles have a great affinity for positive charges. Further consideration of what this affinity may be is not given, but Franck's work on the mobility of ions in A is cited. Here the negative ion seems to exist as an electron; i. e., is not associated with A mol., while the positive ions show a normal mobility. In expts. by W. on smoke and dust between parallel electrodes, the air being ionized by means of X rays, the curve showing the relation of the velocity with which the smoke particles fell under the influence of various fields trengths was just like the curve of the ionization current, so that if the elec. field was strong enough to produce a satn. current, then increasing the field did not increase the rate at which the smoke fell. This is in agreement with Schmidt's observation that the quantity of active deposit which accumulates on the cathode bears the same relation to the elec. field as does the current in the gas.

Radium Preparations (FISCHER). 17.

## 4. ELECTROCHEMISTRY.

C. G. FINK.

Large Electric Furnace. A. HELFENSTEIN. Z. Elektrockem., 17, 642.—No details are given. The furnace is of the enclosed type and particularly adapted for the production of pig Fe and for Zn. Successful runs of over a year's duration have been made with furnaces of 5000-12000 h. p.

C. G. F.

The Effect of Power Factor and Load Factor in Electric Furnace Work. J. HARDEN. Electrician, 67, 713.—Neither the induction furnace, as such, nor the plain are furnace work under ideal conditions, the former because of its lower power factor and the latter because of its unsteady load. Therefore, a furnace so designed as to combine the good features of both, i. e., the higher power factor of the arc furnace and the high and steady load factor of the induction furnace—would undoubtedly be very desirable, provided

the design be simple and reliable. The "Paragon" furnace, recently patented, is designed on the above principles.

C. F. YESSLER.

The World's Production of Aluminium. Anon. Iron Age, 88, 507.—A total of 34,000 metric tons were produced in 1910 compared to 11,500 in 1905. The industry is in the hands of 12 companies. The price in 1910 ranged from 11.9 to 15.5 cents per lb.

C. G. F.

Rapid Changes in the Aluminium Industry. W. C. PHALEN. Mining Sci., 64, 274-5.

J. J. M.

Electrical Resistance of Pure Metals at Liquid Helium Temperatures. H. KAMERLINGH ONNES. Electrician, 67, 657.—Liquid He is an excellent insulator. The resistance of pure Au was not appreciable at the b. p. of He. The resistance of pure Hg was found to be much smaller at the b. p. of He than at H temps.; that the resistance at that stage was not independent of temp.; and at very low temps, such as could be obtained by He evapg. under reduced pressure, the resistance was nil. The resistance first became measurable at slightly more than 4.2° K., at which the resistance was found to be 230 microhms or 1/100,000 of the resistance (solid) at 0°.

C. F. YESSLER.

The Indications of Hot-wire Instruments with Rapid Oscillations. W. STEINHAUS. Kiel. Physik. Z., 12, 657-9.—The newer types of these instruments are reliable and convenient for frequencies up to 10,000 per sec. The errors do not average over 1% and are independent of the frequency.

W. P. WHITE.

A Method of Measuring the Fluctuations in a Rapidly Varying Resistance. F. C. Brown and W. H. Clark. Iowa State Univ. Physic. Rev., 33, 53-9.—The lack of balance of a Wheatstone's bridge was measured with a ballistic galvanometer.

W. P. WHITE.

The Expression of Resistivity of Electrical Conductors. J. H. Dellinger. Electrician, 67, 708.—The author discusses the desirability of expressing resistivity as "mass resistivity" rather than vol. resistivity, the chief reasons being that the measurements of either cross-section or d. in many cases is difficult and inaccurate and conductors are sold by wt. rather than by vol., and, therefore, the information of value to most users is given directly by the "mass resistivity." The use of the term "resistivity" is advocated in place of "sp. resistance." Also limitations are placed on the use of the term "% conductivity."

C. F. Y.

Temperature Coefficients of Electrical Resistance. A. A. SOMERVILLE. Cornell Univ. Physic. Rev., 33, 77-80.—Continuing previous work (C. A., 4, 3039), the coeffs. of various interesting metals or useful alloys are given. Sn and Zn show over 100% increase on m. Melting Zn has a coeff. of nearly zero in a quartz tube, but held together in the air by surface tension it has a negative coeff. Si decreases from 0° to 220° about 25%, increases to nearly the original value by 500°, then decreases again nearly to half value at 700°. Of com. alloys, at 800° brass has added 140% of the resistance at zero; Climax and Kruppite, 40%; phosphor bronze and cupror, 80 and 100%; German silver, 15%; monel metal 10%, but up to 350° the monel has only about 1/4 the coeff. of German silver. At 450°, monel has only increased 3%, and the increase is proportionately less at lower temps.

W. P. White.

The Formation of Hydrocyanic Acid in the High Potential Arc. A. V. LIPINSKI. Zurich. Z. Elektrochem., 17, 761-4.—The reaction investigated is:  $2CH_4 + N_9 = 2HCN + 3H_2$ . To keep C from depositing on the Pt electrodes, H<sub>2</sub> and an excess of N<sub>2</sub> were added. The glass reaction bulb had a capacity of 3.8 l. A current of 4200 volts was used. The O<sub>2</sub> (impurity) content of the reaction mixt. varied between 0.5

and 0.1%. The best output was 19% HCN by vol. and was obtained from a mixt. of 20 parts CH<sub>4</sub>, 10 H<sub>2</sub>, and 70 N<sub>2</sub>.

L. K. MULLER.

The Production of Ozone. C. HARRIES. Kiel. Z. Elektrochem., 17, 629-33.-The author obtained a max. output of O<sub>2</sub> of 18-19% at 20° and 7400 v. a. c., using pure O<sub>2</sub>. The app. was a 10-tube Siemens and Halske ozonizer with a wooden supporting frame. The max. efficiency was attained at a velocity of 60 l. per hr. for parallelconnected and 8.6 l. per hr. for series-connected tubes. The current frequency was 100 cycles per sec. The efficiency of the app. increases after repeated use. Pure O, when passed through the tubes did not give rise to luminescence; an intensive blue was obtained with air or pure N. The blue constituent of ordinary O, could not be femoved with KOH soln., consequently the presence of blue oxides of N seems improbable (cf. Warburg and Leithaeuser, C. A., 4, 1934). The new products, ethylene ozonide, CH<sub>2</sub>O<sub>2</sub>CH<sub>2</sub>, and propylene oxozonide, CH<sub>2</sub>CHO<sub>4</sub>CH<sub>2</sub>, are comparatively stable, highly explosive oils of a penetrating odor, b. in vacuum without decomp. O<sub>2</sub> reduces to O<sub>2</sub> when brought into intimate contact with satd. organic compds., i. c., CH<sub>2</sub>(CH<sub>2</sub>)<sub>x</sub>- $CHO + O_2 = CH_2(CH_2)_2CHO_2 + O_2$ . By repeated alternate b. and condensing (using liquid-air cooled condensers) an 80% pure O2 was obtained. This product was liquefied and then slowly boiled; the products were collected in 2 large glass bulbs, connected in series and previously carefully evacuated. The titrimetric KI value of the bulb nearest the O, vessel was invariably higher than the O, value obtained by direct weighing of the bulb; in the case of the other bulb the values agreed with each other. The author concludes that O<sub>4</sub> is present in O<sub>2</sub>; this having a higher b. p. would tend to accumulate in the bulb nearest the O<sub>2</sub> vessel. Further details are to be published shortly. In the discussion of the paper, E. H. Riesenfeld reported that he had found the reaction to be  $3SO_2 + O_3 = 3SO_3$  and not  $SO_2 + O_3 = SO_3 + O_3$ ; cf. Trans. Roy. Soc. London, (A) 162, 435 (1872).

The Action of Ozone on Air. M. O. TROY. Chem. Eng., 14, 321.—O<sub>3</sub> may well be used in the treatment of air for the destruction and removal of noxious odors, organisms, and emanations. 60 liters of air containing 13 mg. of ozone will disinfect 324 cu. m. of air but in most cases it will depend on the nature and condition of purity of the air. Expts. showed that germs failed to grow in air which contained 0.6-1% O<sub>3</sub> by vol., while for deep-seated germs about 5% is needed. G. F. YESSLER.

Electrolytic System of Amalgamating Gold Ores. E. E. CAREY. Chem. Eng., 13, 190; Mining Sci., 63, 390-2; Mining World, 34, 889-91; see C. A., 5, 1022.

C. G. F.

Mechanics of the Edison Battery. F. H. CALVIN. Am. Mach., 35, 241.—Details of the mechanical parts.

C. G. F.

Manufacture of Drawn Tungsten Filament Lamps. Anon. Engineering, 92, 233-4; Electrician, 67, 779.—A brief account. No details of manuf. are given.

C. G. F.

History of Incandescent Lamp Manufacture. H. Schroeder. Gen. Elec. Rev., 14, 426-31. C. G. F.

Melting Point of Tantalum (PIRANI, MEYER). 2.

Quick Quenching Spark Caps for Close Coupling (BOAS). 2.

Ozone Plant of St. Petersburg (ERLWEIN). 14.

Barlow, C. W.: Magnetism and Electricity. University Tutorial Press. Thompson, M.: Applied Electro-Chemistry. New York: Macmillan. 8°.

Brit., 8,031, Apr. 2, 1910. GEN. ELEC. Co., Schenectady, N. Y. A conductor of ductil tungsten which retains its ductility after prolonged heating is obtained by producing metallic W in the form of a relatively coarse powder by packing a fused quartz tube with pure WO<sub>3</sub> and reducing the oxide by heating the tube to 1100–1400° while pure dry H is passed through it for 5–15 hrs. or longer.

Ger., 235,914, Nov. 11, 1909. A. R. LINDBLAD and O. STALHANE. Obtaining zinc in elec. shaft furnaces: (see C. A., 5, 2371).

Ger., 236,091, Apr. 22, 1910. W. GÜNTHER, Cassel. In the precipitation of copper from its solns. by means of Fe scrap, passing a current from time to time, with the Fe scrap as cathode, for the purpose of producing a porous Cu layer during the passage of the current, while during the intervals the soln. of the Fe proceeds in the known manner.

Ger., 236,882, Sept. 2, 1905. I. Moscicki, Freiburg, Switzerland. App. for the treatment of gases or gas mixtures with the electric arc. The arc is formed between an inner and a surrounding electrode under the influence of a magnetic field. A displacement of the surface of the arc, which would result in a second heating of the gas mixt., does not occur, because the inner electrode terminates within the outer electrode, and the introduction of the reaction gas is effected along the inner electrode towards its free end, in consequence of which the arc curves from the inner electrode in a closed curve. The yield of N oxides is very great.

## 5. PHOTOGRAPHY.

LOUIS DERR.

Advances in Photochemistry and Photography in 1910. E. VALENTA. Chem. Ztg., 35, 962-3.

J. J. M.

Review in the Field of Photochemistry and Photography in 1910 (I). E. VALENTA.

Chem. Ztg, 35, 941-3.

J. J. M.

The Preparation of Neutral-gray Cells and Layers for Photographic Purposes. Goldberg. Leipsic. Chem. Ztg., 34, 964.—A lampblack emulsion in gelatin may be readily prepared to give any desired absorption coeff. and may be advantageously used in photometric and sensitometric work in place of the neutral-gray glass. The lampblack should be free of fat. Such emulsions are much cheaper and universally more satisfactory.

E. J. WITZEMANN.

Development and Development Factors. R. RENGER. Phot. Rundschau, 25, 202-4.—The development factors obtained by Watkins for the various developers are plotted graphically. The time in seconds is taken from the emersion of the plate in the developer until the first visible part of the image appears. By means of the graph the time required to complete the development of the negative is easily determined.

W. B. H.

PLOTNIKOV: Photochemische Versuchstechnik. Leipzig: Akademische Verlagsgesellschaft. 12 M.

#### 6. INORGANIC CHEMISTRY.

H. I. SCHLESINGER.

Remarks on the Atomic Weight Determinations of Gold. E. PRIWOSNIK. Oesterr.

Z. Berg-Hattenw., 29, 391-5.—Chiefly a review of Berzelius' classical work described in the Stockholm Roy. Acad. of Science in 1844.

E. J. ERICSON.

Recent Progress in the Knowledge of the Rare Barths. R. J. MEYER. Berlin. Z. Elektrochem., 17, 633-42.—A review. The individual rare elements differ more widely from one another in their magnetic behavior than in any of their other properties. This the only element utilized on a large scale; the yearly world consumption is 400,000 kg. In discussion, G. Tammann and Vogler reported that Ce reacted and combined very readily with Pb and Sn.

C. G. Fink.

Action of Sodium Hyposulfite on Copper Sulfate in Aqueous Solution. J. B. FIRTH AND J. E. MEYERS. Univ. of Manchester. J. Chem. Soc., 99, 1329-33; Proc. Chem. Soc., 27, 139.—At ord. temp. in an atm. of CO<sub>2</sub> (Firth and Myers, Proc. Chem. Soc., 27, 96) an acidified mixt. of CuSO<sub>4</sub> and NaH<sub>2</sub>PO<sub>2</sub> solns. passes through a series of changes, yielding unstable products which seem to be Cu<sub>2</sub>O, Cu<sub>2</sub>H<sub>2</sub> and Cu phosphate. On standing, H is evolved and Cu pptd., in part forming a mirror. At about 70°, the same solns. produce, after about 2 min., nearly pure Cu<sub>2</sub>H<sub>3</sub>, later Cu and H. The dry Cu<sub>2</sub>H<sub>3</sub> decomposes slowly in moist air at ord. temp. Sudden decomp. occurs at about 60° with production of H and spongy Cu. It appears that at ord. temp. the production of H precedes that of the hydride while at 70° the order is reversed.

D. M. LICHTY.

The Action of Nitrogen on Lithium Carbide. S. A. TUCKER AND H. R. MOODY. Columbia Univ. J. Am. Chem. Soc., 33, 1478-85.—The absorption reaches a max. at 925° and in 1 hr. The most efficient pressure is 50 lbs. per sq. in. The proportion of N<sub>2</sub> fixed does not differ greatly from that in "nitrolime" but the time required is much less. The nature of the N compds. formed appears to differ considerably from those formed when CaC<sub>2</sub> is used.

A. R. MIDDLETON.

The Atomic Weight of Tantalum. WILLIAM H. CHAPIN AND EDGAR F. SMITH. Univ. of Penn. J. Am. Chem. Soc., 33, 1497-1504.—From the mean of 8 detns. of the ratio TaBr<sub>5</sub>: Ta<sub>3</sub>O<sub>5</sub>, Ta = 181.80. Evidence for the existence of TaOBr<sub>3</sub> is presented.

A. R. MIDDLETON.

The Action of Hydrofluoric Acid Gas upon Certain Oxides. WALTER K. VAN HAAGEN AND EDGAR F. SMITH. Univ. of Penn. J. Am. Chem. Soc., 33, 1504-6.—The results of a large number of expts. on oxides, minerals and salts are detailed.

A. R. MIDDLETON.

The Action of Salicylic Acid upon the Metallic Acids. John Hughes Moller. Univ. of Penn. J. Am. Chem. Soc., 33, 1506—10.—Cb and Ta are quant. pptd. from alk. soln. in absence of alk. fluorides. Ti forms an intensely yellow soln. Attempts to develop a colorimetric estn. of Ti by this soln. were not entirely successful, owing to interference of the common contaminants of TiO<sub>2</sub>. W and Mo give no ppts. Mo in quantity completely prevents pptn. of Cb.

A. R. MIDDLETON.

Potassium Barium Orthosulfarsenate. (KBaAsS<sub>4</sub> + 6 H<sub>2</sub>O), (K<sub>2</sub>AsS<sub>4</sub> + Ba<sub>2</sub>Aa<sub>2</sub>S<sub>3</sub> + 18 H<sub>2</sub>O) EMANUEL GLATZEL. Stadt. Oberrealschule, Breslau. Z. anorg. Chem., 71, 209–14.—KBaAsS<sub>4</sub> + 6 H<sub>2</sub>O, prepared from equal parts of Ba<sub>2</sub>As<sub>2</sub>S<sub>3</sub>.6H<sub>2</sub>O (C. A., 5, 1883) and KCl, forms transparent, colorless kernels which turn white or yellow in the air are easily sol. in H<sub>2</sub>O, are decomp. by acids with formation of As<sub>2</sub>S<sub>3</sub>, and change into a yellow powder with loss of H<sub>2</sub>O when heated in a closed tube.

GEORGE W. MOREY.

Periodides and Perbromides of the Alkaline Earth Metals. W. HERZ AND ALFRED BULLA. Pharm. Inst., Univ. Breslau. Z. anorg. Chem., 71, 254-6.—Solns. containing known amts. of BaI, were treated with varying amts. of I soln. and shaken with CCl. Knowing the partition coeff. of I between H<sub>2</sub>O and CCl. (85.5) the amt. of free I was

calc. from the amt. extracted by the  $CCl_4$  and the total I in the aq. layer. From these data the value of K in  $(BaI_2)(I_2)/(BaI_4)$  was calc. and in 5 expts., using 1.054 millimol. BaI<sub>2</sub> and 0.0168-0.1099 millimol. I<sub>2</sub> per 10 cc., the values obtained varied irregularly from 0.00653 to 0.00753. With higher I conc. K was a little lower, probably due to formation of higher periodides. With  $CaI_2$  (1.0814 millimol. per 10 cc.) and I (0.01188-0.08118 millimol. per 10 cc.), K ranged from 0.00815 to 0.00756, decreasing with increasing I content. Using  $SrI_2$  (1.246 millimol) and I (0.01832-0.0792 millimol. per 10 cc.) K fell from 0.0101 to 0.00949. The alk. earth bromides show the same tendency to form perbromides, but the dissociation const. is larger. Using  $BaBr_2$  solns. (0.9936 millimol. per 10 cc.) and Br solns. (0.00544-0.03267 millimol.), K = 0.314-0.445. With  $CaBr_2$  (1.014 millimol. per 10 cc.) and Br solns. (0.00346-0.02425 millimol.), K = 0.392-0.426.

The Combining Power of the Chlorides of Cu, Pb, Fe, Zn, Sn and Bi and the Combining Power of the Chlorides, Bromides, and Iodides of Cu and Cd and the Sensibility of their Mixed Crystals to Light. GOTTFRIED HERRMANN. Physik.-Chem. Inst., Univ. Göttingen. Z. anorg. Chem., 71, 257-302.—The various salt pairs were studied by fusing the components in Jena glass, and taking cooling curves, using a Pt-Pt-Rh thermocouple. The vol. of the salts taken was always the same. When possible the cooled reguluses were studied microscopically, but it was not possible to prepare specimens in mixts. containing ZnCl, FeCl, SnCl, BiCl, and BiBr. In the system PbCl,-Cu<sub>2</sub>Cl<sub>2</sub> the 2 components are completely immiscible in the solid phase. The m. p. of PCl, was detd. as 501°, that of Cu<sub>2</sub>Cl<sub>2</sub>, 424°. The eutectic comp. is 34% Cu<sub>2</sub>Cl<sub>2</sub>, The system PbCl<sub>2</sub>-CdCl<sub>2</sub> (m. p. 588°) is similar, with an eutectic at m. p. 280°. 30% CdCl, and 385°. The eutectic in the system PbCl<sub>2</sub>-FeCl<sub>2</sub> (m. p. 298°) is at 50% FeCl, and 178.6°. In the system PbCl<sub>2</sub>-SnCl<sub>2</sub> (m. p. 247.2°) a complete series The system Cu,Cl,-SnCl, of mixed crystals is formed, with an intermediate m. p. belongs to the ordinary type, with an eutectic at 77.5% SnCl<sub>2</sub> and 172°. Cu<sub>2</sub>Cl<sub>2</sub> and ZnCl<sub>2</sub> (m. p. 261.5°) form mixed crystals within the limits 0-18 and 95-100% ZnCl<sub>2</sub>. The eutectic is at 90% ZnCl, and 243°. Pure ZnCl, supercooled 4°. The fusion curve of Cu<sub>2</sub>Cl<sub>2</sub> and BiCl<sub>2</sub> (m. p. 224°) is of the ordinary type, with an eutectic at 190° and Pure BiCl, supercooled 9°. In the system Cu,Cl,-FeCl, the components are completely miscible in the liquid phase, completely immiscible in the solid phase, and a compd., Cu<sub>2</sub>Cl<sub>2</sub>(FeCl<sub>2</sub>)<sub>2</sub> (m. p. 320°) is formed. The Cu<sub>2</sub>Cl<sub>2</sub>-Cu<sub>2</sub>Cl<sub>2</sub>.(FeCl<sub>2</sub>)<sub>2</sub> eutectic is at 304° and 50% Cu<sub>2</sub>Cl<sub>2</sub>, the Cu<sub>2</sub>Cl<sub>2</sub>(FeCl<sub>2</sub>)<sub>3</sub>-FeCl<sub>2</sub> eutectic at 263° and 88% Cu<sub>2</sub>Cl<sub>2</sub>. The compd. Cu<sub>2</sub>Cl<sub>2</sub>(FeCl<sub>2</sub>)<sub>2</sub> + 8 H<sub>2</sub>O was prepared by dissolving the components in mol. proportions in H<sub>2</sub>O slightly acid with HCl and letting stand some time, when clear green needles are deposited. Solns. of FeCl<sub>2</sub> dissolve Cu<sub>2</sub>Cl<sub>2</sub>, in the mol. proportion of 2:1. The system ZnCl,-SnCl, belongs to the simplest type, with the eutectic at 171° and 64% SnCl<sub>2</sub>. The system BiCl<sub>2</sub>-FeCl<sub>2</sub> is of the same type, with an eutectic at 171.5° and 23% FeCl<sub>2</sub>. FeCl<sub>3</sub>-ZnCl<sub>2</sub> show partial miscibility in the solid phase, mixed crystals being formed from 0-15 and from 93-100% ZnCl<sub>2</sub>. The eutectic is at 214° and 70% ZnCl<sub>2</sub>. The system CdCl<sub>2</sub>-SnCl<sub>2</sub> belongs to the common type, with an eutectic at 229° and 90% SnCl<sub>2</sub>. The fusion diagram of the system CdCl<sub>2</sub>-ZnCl<sub>2</sub> shows the limiting case of the ordinary type, the eutectic being at 262° and 100% ZnCl<sub>2</sub>. The system PbCl<sub>2</sub>-ZnCl<sub>2</sub> is similar, but on account of pronounced supercooling the thermal effects are indistinct. BiCla-ZnCla are only partially miscible in the liquid phase, the limits being at 12 and at 97.5% BiCl, the latter being un-The eutectic in the system PbCl<sub>2</sub>-BiCl<sub>3</sub> is at 219° and 90% BiCl<sub>3</sub>. from 0-70% BiCl, show a break in the cooling curve at 323.5°, but the comp. of the compd. formed could not be detd. The system PbBr<sub>2</sub>-BiBr<sub>2</sub> is very similar. PbBr<sub>2</sub> m. 380°, BiBr<sub>2</sub>, 219°. An eutectic is formed at 80% BiBr<sub>3</sub>, which crystallizes at 205.3°.

The PbBr, crystals react with the melt of comp. 64% BiBr, at 238-5°, but the comp. of the compd. could not be detd. Cu<sub>2</sub>Cl<sub>2</sub> and CdCl<sub>3</sub> form a continuous series of mixed crystals, the fusion curve showing minimum at 410° and 20% CdCl<sub>2</sub>. In mixts. of 20-60% CdCl<sub>2</sub> a transformation takes place, with a max. of 350° at 30%, CdCl<sub>2</sub>, corresponding to the compd. (Cu<sub>2</sub>Cl<sub>2</sub>)<sub>2</sub>CdCl<sub>2</sub>. Sections of reguluses containing from 10-90% Cd, at first transparent, become cloudy and gray-blue color, under the action of light. The development of the gray-blue color seems to be associated with the formation of (Cu<sub>2</sub>Cl<sub>2</sub>),CdCl<sub>2</sub>, being a case of photosynthesis in which a colored compd. is formed by the action of light on the mixed crystals. In the system Cu<sub>2</sub>Br<sub>2</sub> (m. p. 484°), CdBr<sub>2</sub> (m. p. 568°) mixed crystals are formed from 0-66% CdBr<sub>2</sub>, where a fusing point minimum at 422° is formed. From this point pure CdBr, is formed, the fusion curve rizing to the m. p. of CdBr<sub>2</sub>. A transformation takes place from 394-374°, the temp. decreasing with increasing conc. of CdBr<sub>2</sub>. In this case also the mixed crystals react under the influence of light, with change of color, and probable formation of Cu<sub>2</sub>Br<sub>2</sub>(CdBr<sub>2</sub>)<sub>2</sub>. Cu<sub>2</sub>I<sub>2</sub> (m. p. 606.5°) and CdI<sub>2</sub> (m. p. 392.4°) form a continuous series of mixed crystals, with a minimum in the curve at 90% CdI<sub>2</sub> and 350°. Pure Cu,I, shows a transformation at 414°, the temp. of which is lowered by addition of CdI, and which is still perceptible at 95% CdI. All reguluses containing Cu<sub>2</sub>I, change from clear yellow to dark brown under the influence of light. The author finds no parallel between the combining power of metals and their salts, the latter showing much less tendency to form compds. than the former. He notes that the property of supercooling is a distinctive property of some salts, and is retained in its mixts.

G. W. M.

Action of Ammonia on Mercurous Nitrate. HARIDAS SAHA AND KUMUD NATH CHOUDHURI. Chem. Lab. Docca Coll. Eastern Bengal and Assam, India. Z. anorg. Chem., 71, 309-12.—The black ppt. obtained from HgNO<sub>3</sub> and NH<sub>4</sub>OH is a mixt. of Hg and a white substance of the empirical formula N<sub>2</sub>Hg<sub>2</sub>H<sub>4</sub>O<sub>4</sub>. This latter substance is sol. in excess of NH<sub>4</sub>OH, insol. in KOH, is stable at 100°, and decomposes on heating.

G. W. M.

Compounds of Silver with Cadmium. G. I. PETRENKO AND A. S. FEDOROV. Phys.-Chem. Lab., Univ. Kharkov. Z. anorg. Chem., 71, 215-8; cf. C. A., 5, 1734.—If the compd. AgCd<sub>4</sub> (m. p. 530°) exists, when reguluses of that compd. are heated for a long time at 510-20° they should show only the characteristic polygonal structure under the microscope. Actually, however, both large white crystals containing inclusions and inhomogeneous small crystals were observed, the relative amts. of which varied with different heat treatments. From this the authors conclude that the compd. AgCd<sub>4</sub> does not exist. Similar expts. with AgCd<sub>4</sub> showed a polygonal structure, which makes more probable the existence of that compd. G. W. M.

New Formulas for Preparing a Mixture of Fuming Sulfurio Acid of Given Strength. José Prats Aymerich. Barcelonia. Anales soc. españ. fis. quim., 8, 78-83.—For prep. of mixts. of ordinary and fuming  $H_2SO_4$  of given  $SO_2$  content for sulfonation, etc., the following formula is proposed: it has the advantage over Guehm's formula of not requiring the use of tables and of being adapted without further calc. to any given wt. of fuming  $H_2SO_4$ . If a = wt. of fuming  $H_2SO_4$  to be prepared (with a content of h% of  $SO_3$ ), x = wt. of fuming  $H_2SO_4$  (containing h% of  $SO_3$ ) required for the mixt. and y = required wt. of ordinary  $H_2SO_4$  (of s% purity), then: (1) x = a[9h + 40(100 - s)]/[9k + 40(100 - s)]; (2) y = a - x = a9(k - h)/[9k + 40(100s)]. If ordinary  $H_2SO_4$  of sp. gr. 1.840 (= 95.60% pure) be used and the approx. value 4.5 be taken for 100 — s, formulas (1) and (2) become: (3) x = a(h + 20)/(k + 20); (4) y = a(k - h)/(k + 20). Formulas (1) and (2) are accurate and (3) and (4) are accurate

enough for ordinary use. Calculations are given comparing these 2 sets of formulas with each other and with Guehm's formula.

H. S. PAINE.

Action of Carbon on Chromyl Chloride. José R. Mourelo and Antonio G. Banús. Lab. quim. inorgán. orgán. ind., Escuela Ind., Madrid. Anales soc. españ. fis. quim., 8, 355-62.—With the expectation of obtaining carbonyl chloride according to the equation  $4\text{CrO}_2\text{Cl}_2 + 4\text{C} + O_2 = 2\text{Cr}_2\text{O}_2 + 4\text{COCl}_3$ , air (dried by H<sub>2</sub>SO<sub>4</sub> and P<sub>2</sub>O<sub>4</sub>) was passed through a Winkler spiral containing CrO<sub>2</sub>Cl<sub>2</sub> (heated by a water bath) and then through a combustion tube containing well dried charcoal heated to redness. Carbonyl chloride was not formed, however, and, when CrO<sub>2</sub>Cl<sub>2</sub> was in excess, Cr<sub>2</sub>O<sub>4</sub>, CrCl<sub>4</sub>, and a white sublimate were formed in the tube in the order named. The C used above was that of ordinary charcoal crayons; even after drying for 2 hrs. at 150° there was abundant formation of HCl when it was heated to redness and Cl passed over it. When the amt. of CrO<sub>2</sub>Cl<sub>2</sub> was diminished (by lowering the temp. of the water bath containing the Winkler spiral) and the reaction allowed to take place in an atm. of Cl (charcoal obtained from the wood of the rock-rose was used here), a sublimate of CrCl<sub>2</sub>, but no Cr<sub>2</sub>O<sub>4</sub>, was obtained.

H. S. Paine.

Absorption of Carbon Dioxide by Soda Lime. José Casares. Anales soc. españ. fis. quim., 9, 198-201.—Expts. show that, contrary to the usage recommended by many text-books on analytical chem., CO, is not absorbed by perfectly dry soda lime and that a certain amt. of moisture is necessary for complete absorption. H. S. Paine.

The Action of Iodine on Chlorates and Bromates. KLINGER. Koenigsberg Chem. Ztg., 34, 1023.—There is no simple reaction to explain this action. By the attack of I on the chlorate (even c. P. I reacts with c. P. KClO<sub>3</sub> without the addition of acid) Cl is evolved, this acts on I giving ICl<sub>3</sub> and this again acts on chlorate; when ICl<sub>3</sub> acts directly on the chlorate the iodate is obtained at once. The HClO formed at first decomposes into Cl and HClO<sub>3</sub> as the temp. rizes. With Ba and Ca salts this reaction proceeds very smoothly either to form the chloride and HClO or Cl and HClO<sub>3</sub> according to the temp. The action of I on bromates is simpler. Br and neutral iodate are formed at once. KI was used in place of I but the reaction did not proceed well when more than 1/2 of the I was used as KI. Some I must always be present at the beginning to produce the reaction at all.

E. J. WITZEMANN.

Complex Salts: Pyridinopentachloro-iridites. MARCEL DELÉPINE. Bull. soc chim., 9, 710-7; cf. C. A., 5, 2605, 2787.—A somewhat more detailed presentation, giving analytical data and description of individual salts.

EDW. WOLESENSKY.

Method of Reduction in Presence of Finely Divided Palladium. P. BRETEAU. Bull. soc. chim., 9, 764-70.—In the presence of 5 g. of freshly prepared Pd-black and in contact with pure H, 10 g. of phenanthrene in 150 g. of cyclohexane was reduced to the tetrahydride of phenanthrene. The dihydride was not detected in any case. The same results were obtained by reducing phenanthrene in alc. soln. by means of H in the presence of pptd. Pd. The H in this case was obtained from the interaction of a metal and HCl, both of which were added directly to the above soln. Similar results were also obtained by the electrolysis of an alc. soln. of phenanthrene containing a little H<sub>2</sub>SO<sub>4</sub>, using a cathode covered with Pd sponge and a current of about 10 amp. under 8 volts.

Action of Thionyl Chloride on Metallic Oxides. G. Darzens and F. Bourion. Compt. rend., 153, 270-2.—SOCl<sub>2</sub>, as a chlorinating agent, behaves like a mixt. of Cl and S<sub>2</sub>Cl<sub>2</sub> not only in the character of the products but also in the rate of reaction and the temps. at which the reactions begin. The same relation is brought out in a study of the heats of reaction. It is, however, inferior to a mixt. of Cl and S<sub>2</sub>Cl<sub>2</sub> or to S<sub>2</sub>Cl<sub>3</sub> alone, since it decomposes above 440°, is more difficult to prepare, and for analytical

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purposes must be carefully freed from P compds. which with H<sub>2</sub>O would yield the non-volatil P<sub>2</sub>O<sub>3</sub>.

EDW. WOLESENSKY.

Andersen, J. H.: Darstellung von Calcium und Calciumlegierungen. Berlin: E. Ebering. 8°, 35 pp., 1 M.

HOOTON, W. M.: Inorganic Chemistry for Schools. London: E. Arnold. 8°, 414 pp., 3 s. 6 d.

LICKFETT, H.: Chloride, Bromide and Fluoride des Vanadins. Berlin: E. Ebering. 8°, 67 pp., 1.50 M.

MEYER, F.: Neubestimmung des Verhältnisses des Molekulargewichtes von Kaliumchlorat und Kaliumchlorid. Berlin: E. Ebering. 8°, 47 pp., 1.30 M.

## 7. ANALYTICAL CHEMISTRY.

#### E. G. R. ARDAGH.

Analysis of Lead Bronzes and Brasses. Q. Sestini. Lab. chim. ind. r. ist. tech. Bergamo. Ind. chim., 11, 229-31.—By employing a slightly modified form of the Hollard app., the sepn. of Cu and Pb can be effected much more simply than by the Classen method and with just as great accuracy. To the exterior of the anode are attached little sheets of Pt, polished with very fine emery, to increase the adherence of the PbO<sub>2</sub> and the surface of the anode. The soln., strongly acid with HNO<sub>2</sub> and heated to 70°, is electrolyzed 1 hr., then NH<sub>2</sub> added in such amt. that 5 cc. of HNO<sub>2</sub> remains free and the electrolysis continued until the deposition of Cu is complete (3-4 hrs.). A single weighing will give the amt. of Cu and Pb. The Sn is first pptd. as metastannic acid, and after the electrolysis the Fe is pptd. as Fe(OH)<sub>2</sub> and the Zn deposited electrolytically on the cathode on which the Cu has been previously deposited after (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> and a slight excess of H<sub>2</sub>SO<sub>4</sub> have been added to the ammoniacal soln. The analysis can be completed in a single day.

Modifications of the Arnold and Hardy Method for the Rapid Quantitative Determination of Sulfur in Siderurgical Products. F. GIOLITTI AND M. MARCANTONIO. Lab. chim. metallurg. e metallogr. r. polit. Torino. Rass. min., 35, 67-9.—To prevent the loss of S in the form of org. compds. or by imperfect absorption of the H<sub>2</sub>S, the Arnold and Hardy method (Chem. News, 1888, No. 1496) has been modified as follows: The gases (H<sub>2</sub>S, H, HCl, org. S compds., etc.) issuing from the decomp. flask pass through a short condenser and a hard glass tube heated to redness (to decomp. the org. compds.) into a train of absorption bulbs containing neutral Pb or Cd acetate and kept at a temp. of 70-90°. These bulbs consist of pear-shaped vessels; through 1 side, near the top, passes a tube, extending nearly to the bottom, through which the gases enter and, after bubbling through the soln., they pass out through another side tube connected with the entrance tube of the next bulb. The results on samples of iron and steel containing 0.011-0.259% S were 0.001-0.004% lower than those obtained by oxidizing and weighing as BaSO<sub>4</sub>.

C. A. R.

A Modification of the Oxidation Method for Determining Sulfur. WARREN I. KEELER. Iron Age, 86, 492-3.—The evolution method for the detn. of S in Fe and steel, in spite of its drawbacks, is used in most foundry and steel works labs. The author uses the following modification of the oxidation method and claims that 2 or 3 accurate detns. may be concluded in 1 hr. Method for Fe: Add 30 cc. conc. HNO, to 2.750 g. sample and warm until soln. is complete, add 5 cc. conc. HCl, evap. to dryness, bake 5 mins. after all nitrous fumes have disappeared, remove from plate, cool, add 20 cc. conc. HCl, evap. to a heavy syrup of about 5 cc., add 2-3 cc. conc. HCl,

bring to the b. p., remove from plate, add 75-100 cc. of hot H<sub>2</sub>O, filter, wash with hot 10% HCl and cold H<sub>2</sub>O, catch filtrate in a tall beaker, add 15 cc. BaCl<sub>2</sub> (20% soln.), slowly bring to b., boil for 15-20 mins., allow to settle for 1/2 hr., filter through double ashless paper, wash the residue with hot dil. acid, allow to run through, refilter and wash alternately with cold H<sub>2</sub>O and hot dil. acid. Method for steel: The above procedure differs only in method of soln. With high C and alloy steel add a little conc. HCl at first and thereafter several times if necessary. Iron Alloys: With ferro alloys, especially ferrosilicon, a small amt. of c. p. HF may be added slowly and cautiously, but in such case the soln. must be baked twice as well or high S result will be obtained.

L. A. TOUZALIN.

Electrolytic Determination of Copper. F. W. TRAPHAGEN. Chem. News, 104, 69-70.—The detailed procedures outlined are those worked out by the Anaconda Copper Mining Co.

H. M. LANCASTER.

A Method for Determining Nitric Acid. O. ANGELUCCI. Chem. Ztg., 34, 994.—The substance (2-5 g.) containing nitrate is mixed with 3 g. fused powdered PbCrO<sub>4</sub> and placed in a porcelain boat in a combustion tube open at both ends, 30 cm. of metallic Cu, 15-20 cm. CuO and 5-6 cm. Cu gauze are put in. This end is then connected with an azotometer and CO<sub>2</sub> passed through. When the air is removed the furnace is gradually warmed to 400-500°. N oxides are reduced by the Cu and any H formed is oxidized by CuO. Results are very satisfactory. E. J. WITZEMANN.

A Reaction for Determining Nitrites. G. Armani and J. Barboni. Chem. Ztg., 34, 994.—Treat 10 cc. of the soln. with 4-5 drops of satd. soln. of benzidine acetate and dil. HOAc. The color ranges from yellow to red depending on the conc. E. J. W.

A Simplified Method for Determining Potassium. A. Grete. Zurich. Chem. Zig., 34, 1040.—After dissolving the substance in a vol. flask add NaOH and NH<sub>4</sub>OH till alk. to phenolph. to ppt. phosphates of Ca, Fe and Al as well as CaSO<sub>4</sub>, fill to mark settle and pipet off an aliquot part, evap. to dryness in a resistance glass beaker first adding additional NaOH to remove NH<sub>3</sub> completely. If amides are present they are composed by adding NaOCl or Cl water. If much organic matter is present the soln. is acidified slightly with H<sub>2</sub>SO<sub>4</sub>, evapd. in a Pt dish, the residue is ignited, then washed into a beaker with a little water and evapd. The residue is acidified with HCl and treated with PtCl<sub>4</sub> in slight excess and evapd. to a few cc. Dissolve in minimum amt. of water and ppt. by adding slowly 100 cc. EtOH 96%, filter off ppt. on Gooch crucible. To remove occluded Na<sub>2</sub>SO<sub>4</sub> dissolve ppt. in water, reduce the Pt with dil. H<sub>2</sub>SO<sub>4</sub> and Mg shavings, filter and proceed as usual. SiO<sub>2</sub> present can be removed by boiling out the Pt with NaOH. Control expts. with pure K salts in mixts. have proved the accuracy of the method.

Report of Referee on Potash. E. L. Baker. Bur. Chem., Bull. 137, 16.—A comparison was made of the official method (Bur. Chem., Bull. 107, 11), a volumetric and a grav. cobaltinitrite method for K<sub>2</sub>O. Drushel's volumetric method (C. A., 3, 36), slightly modified, gave good results by most analysts. The grav. cobaltinitrite method is simpler than the volumetric and promises well, though there is a tendency toward high results. A test was also made of the modification of the official method of making up the potash soln. (Breckenridge, C. A., 3, 2604; 4, 944), washing a weighed amt. of the sample through filter paper with hot H<sub>2</sub>O and detg. K<sub>2</sub>O in the filtrate. This method shows an increase in K<sub>2</sub>O of 0.1-0.3% over the official method.

A. W. BROOMELL.

The Detection of Perboric Acid and Several Similar Compounds. W. LENZ AND E. RICHTER. Pharm. Inst. F. Wilhelms-Univ., Berlin. Z. anal. Chem., 50, 537-44.

—NaBO<sub>2</sub> and K<sub>2</sub>C<sub>2</sub>O<sub>4</sub> give strongly alk. aq. solns. with evolution of H<sub>2</sub>O<sub>2</sub> which may

be removed by acidifying with H<sub>2</sub>SQ<sub>4</sub> and shaking with ether, and proved by sepg. and adding a drop of 1% CrO, soln., obtaining an intense blue color (perchromic acid). (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>2</sub> soln. is acid, yields O and neither the acid soln. nor soln. + NaOH gives H,O, by ether test. KClO, and HIO, give neutral solns. without gas evolution. KMnO<sub>4</sub> reacts with perborates and percarbonates as with H<sub>2</sub>O<sub>5</sub>. (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>5</sub> decolorizes the KMnO<sub>4</sub> as does H<sub>2</sub>O<sub>5</sub>. KClO<sub>4</sub> has no effect in acid soln., but becomes blue after adding NaOH. HIO, has no effect in acid or alk. solns. AgNO, and NaBO, give a heavy black ppt. insol. in 10% NH<sub>2</sub>OH. Perborate soln. acidified with HNO<sub>2</sub> showed no reaction. In soln. + NH4OH a Ag mirror is finally formed. K2C2Oa in aq. soln. gives white ppt., yellowish, then gray, on warming, insol. in warm 10% NH<sub>4</sub>OH but the liquid becomes bluish. Soln. + HNO<sub>2</sub>, no change; soln. + NH<sub>4</sub>OH gives Ag mirror. (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>2</sub> soln. becomes turbid and metallic Ag seps. Soln.+ HNO<sub>a</sub> becomes yellowish; soln. + NH<sub>a</sub>OH evolves gas without sepn. of a ppt. and no change on heating. KClO4, in neutral, HNO3 or NH4OH solns., gives no reaction. Aq. soln. of HIO, gives bright yellow, rhombohedral crystals, readily sol. in NH,OH but insol. in the cold in 20% soln. of Na<sub>2</sub>S<sub>2</sub>O<sub>2</sub> with SO<sub>2</sub>. With excess of NH<sub>4</sub>OH, a slight brownish turbidity results; on warming, a decided brown ppt. MnSO.: A 1% soln. covered with layer of petroleum ether to protect from air was used. NaBO, or K<sub>2</sub>C<sub>2</sub>O<sub>6</sub> in 0.075% soln. gives decided brown. K<sub>2</sub>S<sub>2</sub>O<sub>6</sub> and HIO<sub>4</sub> give same change, but limit of sensibility is 0.025%. KClO4 shows no effect. This test may be made on powdered mixt. of perborates, etc., by placing on slide with a little MnSO<sub>4</sub> soln., covering and examining with a microscope the sepn. of MnO<sub>2</sub> may be easily seen.  $Co(NO_3)_2$ , 0.25% air-free aq. soln. with NaOH (petroleum ether cover), gives blue, then faint red ppt. Two drops of a 1% NaBO, soln., 6 drops of 1% K,C,O, soln., or 1 drop of (NH<sub>4</sub>),S,O, soln. will produce at once a decided brown ppt. KClO4 and HIO4 do not give the reaction. NiSO<sub>4</sub> (1% soln.): Tests applied as with Co(NO<sub>2</sub>)<sub>2</sub>; NaBO<sub>3</sub>, K<sub>2</sub>C<sub>2</sub>O<sub>4</sub>, KClO<sub>4</sub>, HIO<sub>4</sub> gave no reaction. One drop of 1% (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>2</sub> soln. gave a black ppt. This reaction is distinct with 2 drops of a 0.1% soln. of persulfate. H<sub>2</sub>O<sub>2</sub> does not give this reaction. Lead Solution: 40 g. of 15% NaOH are mixed with 10 g. of 10% Pb(OAc), soln. and 50 g. water. This soln. gives, with official H<sub>2</sub>O<sub>2</sub>, a dark ppt. With excess H<sub>2</sub>O<sub>2</sub> a turbidity is produced, which disappears on b. With NaBO<sub>2</sub>, K<sub>2</sub>C<sub>2</sub>O<sub>4</sub>, KClO<sub>4</sub> and small amts. of (NH<sub>4</sub>)<sub>x</sub>S<sub>2</sub>O<sub>8</sub> no reaction is noted. With large amts. of persulfate a yellowred ppt. seps. on heating. HIO4 gives in high dilutions a white, heavy, microcryst. ppt. insol. in NaOH. CeCl<sub>2</sub>: A soln. of 0.2 g. in water with a few drops HCl is used. Two drops of 0.03% H<sub>2</sub>O<sub>2</sub> soln. with 2 drops NH<sub>4</sub>OH and a few drops of the reagent produce a faint yellow; with 0.5 mg. NaBO, and 0.2 mg. K<sub>2</sub>C<sub>2</sub>O<sub>6</sub>, a decided yellow. (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub>, KClO<sub>4</sub> and HIO<sub>4</sub> do not give the reaction. K1: By adding 2 drops of 10% soln. to a 1% aq. soln. of salt to be tested, free I is liberated only with (NH<sub>4</sub>)<sub>s</sub>-S<sub>2</sub>O<sub>2</sub>. On acidifying with dil. H<sub>2</sub>SO<sub>4</sub>, perborate and percarbonate each set I free; KClO4 produces no change; on substituting KBr for KI, in no case could any Br be shaken out with CHCl<sub>2</sub>. Fuchsin: 500 g. fuchsin and 12.5 g. AcONa in 100 cc. water is a reagent for persulfates. Add drop by drop to a few cc. of the soln. being tested and heat. NaBO<sub>2</sub> and K<sub>2</sub>C<sub>2</sub>O<sub>6</sub> readily decolorize and addition of H<sub>2</sub>O does not restore color. AcOH gives yellow. (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>3</sub> decolorizes with difficulty. Excess of H<sub>2</sub>O or addition of AcOH will not restore the color. KClO<sub>4</sub> has no effect. Aniline sulfate: Five g. 20% H<sub>2</sub>SO<sub>4</sub> are diluted to 100 cc., 10 g. pure aniline added and shaken violently. After the aniline has collected in large drops, filter, obtaining a clear, colorless acid soln. Equal parts of this reagent and of 1% solns. of salts were mixed and heated to b. NaBO, and K<sub>2</sub>C<sub>2</sub>O<sub>6</sub> gave very faint yellow. (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub>, a distinct dark brown. KClO<sub>4</sub> remained unaltered. With (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>4</sub> the limit of sensibility is 0.5 mg. All of these mixts., both before and after the expts., turned blue litmus paper red. F. W. SMITHER.

Detection of Methyl Alcohol in Ethyl Alcohol. J. MAYRHOFER. Arch. Chem. Mibros., 4, 183-5.—A brief review of the literature. F. W. SMITHER.

The Determination of Small Amounts of Iodine in Organic Combination. A Modification of Hunter's Method. E. C. Kendall. Path. Dept., St. Luke's Hosp., N. Y. Proc. Soc. Exp. Biol. Med., 8, 120.—The author's modification of Hunter's method (C. A., 4, 431) differs only in the manner of removing Cl. In the original method, after oxidizing the NaI or KI, formed by the fusion, to iodate with NaClO, H<sub>2</sub>PO<sub>4</sub> is added and the excess of Cl removed from soln. by b. The removal of Cl by b. is time-consuming and uncertain. The modification consists in removing the excess of Cl with phenol, which holds the Cl in strong combination and does not interfere with subsequent operations.

V. C. Myers.

Rapid Analysis of Cement (WILEY, EARNST). 20.

Salicylic Acids and Metallic Acids (MULLER). 6.

Reactions for Formation of Iodine Derivatives (GEHARD). 17.

Sulfur in Coal (MUER). 21.

Nitrogen [Determination] (JONES). 15.

Determination of Soluble Phosphoric Acid (WHITEHOUSE). 15.

Methyl-red Indicators (Howard, POPR). 10.

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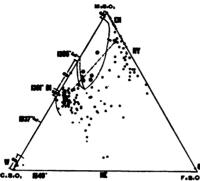
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## 8. MINERALOGICAL AND GEOLOGICAL CHEMISTRY.

#### R. C. WELLS.

Minerals of the Composition  $MgSiO_s$ -CaSiO<sub>3</sub>-FeSiO<sub>3</sub>. R. B. Sosman. Geophys. Lab. J. Wash. Acad. Sci., 1, 54-8.—A study from the statistical standpoint of rocks and minerals having a comp. corresponding to a mixt. of the metasilicates of Mg, Ca and Fe, assuming as seems perfectly justifiable, that they may be treated as a 3-component system. In the accompanying diagram black dots are mineral analyses; circles, rock analyses. EN, enstatite; HY, hypersthene; G, grünerite (amphibole); HE, hedenbergite; W, wollastonite; DI, diopside; E, eutectic; S, solid solution. The following facts are brought out by the diagram: The rock analyses and the mineral analyses can be readily separated by a line, which probably represents the boundary between magma comps. that will form homogeneous solid solns. and magmas that will sep. into 2 different solid solns. between which is a eutectic. The rock analyses lie in the

"eutectic field," the minerals outside of it. Under conditions of complete equil., analyses of minerals from igneous rocks of these comps. should all lie along the border



line of the "eutectic field." If equil. were incomplete during solidification, the comp. of the constituent minerals might lie some distance away from this boundary as some plotted actually do. The diagram also includes many secondary minerals which may have almost any comp. within the solid portion of the diagram. The diagram illustrates very well how it may become possible to classify minerals and rocks on a physicochem. basis, starting with chem. complexity as the fundamental character. Such a classification means more to the chemist than to the mineralogist and petrologist, but it is to be hoped that it will throw increased

light on the relations of the silicates from both points of view. R. C. WELLS.

Quartz and Fluorite as Standards of Density and Refractive Index. H. E. Merwin. Geophys. Lab. J. Wash. Acad. Sci., 1, 59.—The 2 minerals, quartz and fluorite in clear colorless varieties, appear to have d. constant to  $\pm$  0.001 and n constant to  $\pm$  0.001, regardless of locality. These minerals may, therefore, be used as standards. The  $d_{20}$  of quartz is 2.6495 and its  $n_D^{20}$  1.54425. The corresponding values for fluorite are 3.180 and 1.43385.

The Temperature Stability Ranges, Density, Chemical Composition and Optical and Crystallographic Properties of the Alkali Feldspars. H. E. MERWIN. Geophys. Lab. J. Wash. Acad. Sci., 1, 59-60.—Changes in the double refraction of sections of the alkali feldspars, observed during and after heating in the thermal microscope, indicate that both albite and orthoclase appear in 2 forms, the transition temp. of the low temp.  $\beta$ -form into the high temp.  $\alpha$ -form being in both cases about 900°. Sanidine has the optical properties of  $\alpha$ -orthoclase and adularia those of  $\beta$ -orthoclase. Microline appears to be stable to its m. p. Anorthoclase behaves like microcline when heated, except that a slight change in double refraction near 900° has been observed in specimens intergrown with albite. The inversion in both orthoclase and albite is very sluggish even at 1100°. The optic axial angles of sanidines rich in albite are larger than those of pure orthoclase. The presence of albite in orthoclase raises the d., but apparently not so much as might be expected if no vol. change accompanied the mixing; n is generally higher than might be expected under these conditions. As the % of albite increases the length of the crystallographic axis a decreases. The angles in the prism zone and in the zone of the basal and front pinacoids are most affected by this change. Crystals in the same hand specimen often exhibit considerable variation in the value of these particular angles, while other angles remain practically const. Such variations indicate that the different interfacial angles are not equally sensitive to changes in the conc. of albite in the orthoclase. The crystallographic axis c appears to be slightly shorter in adularia than in sanidine. R. C. W.

Crystallized Turquoise from Virginia. Waldemar T. Schaller. Geological Survey. J. Wash. Acad. Sci., 1, 58-9.—A sample of well cryst., bright blue mineral from Campbell Co., Va., proved to be turquoise, a species hitherto known only in a cryptocryst. condition. The minute crystals composing a secondary botryoidal crust on quartz have a rhombic shape and, like the crystals of chalcosiderite, with which turquoise is isomorphous, are triclinic. As the angles of the 2 species are very

close, the crystallographic elements of chalcosiderite are taken for turquoise: a:b:c=0.7910:1:0.6051,  $\alpha=92^\circ58'$ ,  $\beta=93^\circ30'$ ,  $\gamma=107^\circ41'$ . Mean \* 1.63 , double refraction high (0.04) and pleochroism strong, pale blue to colorless. D 2.84. Analysis: P<sub>2</sub>O<sub>4</sub> 29.84, Al<sub>2</sub>O<sub>3</sub> 31.91, Fe<sub>3</sub>O<sub>3</sub> 0.18, CuO 7.87, H<sub>2</sub>O 17.59, insol. 12.57; sum 99.96. Formula, CuO.3Al<sub>2</sub>O<sub>3</sub>.2P<sub>3</sub>O<sub>4</sub>.9H<sub>2</sub>O. R. C. W.

Variscite near Lucin, Utah. L. J. PEPPERBERG. Mining Sci. Press, 103, 233-4.

—The purer specimens of variscite in the Lucin deposit are nodular, oblitic, and brecciated in structure. Variscite has the chem. comp.: AlPO<sub>4</sub> + 2H<sub>2</sub>O or Al<sub>2</sub>O<sub>2</sub>.P<sub>3</sub>O<sub>4</sub>.4H<sub>2</sub>O. The fracture is conchoidal. Tests showed the presence of minute quantities of Fe and Ca and a trace of Cr, and the color ranging from apple-green through the blue-greens is ascribed to the salts of these metals. On intense ignition it becomes white. Variscite is insol. in acids before heating but afterward dissolves readily in acids and alkalies.

E. I. Crane.

Method for Determining the Density of Certain Solids by means of Rohrbach's Solution Having a Standard Refractive Index. H. E. Merwin. Geophys. Lab. J. Wash. Acad. Sci., 1, 52-3.—By means of Rohrbach's soln., so prepared that the relation of its d. to n is known, the d. of suitable solids between 2 and 3.5 can be detd. rapidly and accurately or the total refractometer by finding n of the soln. while fragments of the solid are suspended in it. For the soln. prepared by satg. with HgI<sub>2</sub> at 20° so that its d. is that of ordinary orthorhombic S (2.07), the relation d = 5.39 n = 6.0865 holds for cones. corresponding to ds. between 2.25 and 3.4.

J. JOHNSTON.

Some Gold Deposits of the Northwest. F. Church Lincoln. Eng. Mining J., 92, 408–10.—A résumé of the features of ore deposition in some mines in Montana and Idaho where ore was formed by replacement or by contact metamorphism.

ROBERT KANN.

The Platiniferous Deposits of the Urals. L. DUPARC. Arch. sci. phys. nat., 31, 211-30, 322-45, 439-56, 516-33.—The geological relations of the rocks in which the Pt deposits occur are described in detail and petrographical and chem. data given. Pt occurs chiefly in the dunites, less frequently in the pyroxenites and is often associated with chromite. By 15 analyses of native Pt by H. C. Holtz, ranges in Pt from 75.37 to 88.98; Ir, Os, 0.28 to 5.41; Pd, 0.15 to 0.99; Cu, ;0.03 to 1.66; Rh, Ru, Ir, 2.79 to 3.96; and Fe, 7.03 to 16.60 are shown.

R. C. Wells.

The Tin Deposits of Bolivia. II. M. Armas. Eng. Mining J., 92, 359-63.—In all the lodes Sn occupies preferably either one or both of the walls of the vein and often no Sn is found in the matrix between the walls. If the fissuring is accompanied by rock shattering Sn is found against the false walls so formed; pneumatolytic action, at great depth, under high pressure and temp., explains quite satisfactorily the mode of deposition. The association of Sn deposits with acid rocks is characteristic not alone of Bolivia. Cassiterite veins were formed first, and at later geologic periods the filling of the veins was completed with other mineral matter, pptd. from circulating plutonic water, or they were reopened and refilled at various periods coincident with the upheaval of the mountain range along the coast. Even stannite has been deposited in precedence of the other sulfides. More frequently, however, Sn is found as minute particles of cassiterite encased by sulfides of other metals.

Oxidized Zinc Ores at Leadville. G. O. ARGALL. Eng. Mining J., 92, 399-400. —To a slight extent the oxidized Zn ores are found associated with, or in the immediate vicinity of, the PbCO<sub>4</sub> ores, sometimes forming a more or less complete envelope around the latter. However, by far the greater portion of the Zn has been removed to points more or less remote from the original ore body and redeposited, by a process of replace-

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ment, in depressions in the limestone or along some lower contact plane. The ore consists of smithsonite and calamine, colored reddish to dark brown by the presence of Fe<sub>2</sub>O<sub>5</sub>. The average Zn ore is comparatively free from Pb, though it does contain Fe, Mn and CaO. The Ag content in these ores is low.

R. K.

Comparison of the Waters of the Geysers of Iceland. José Casares. Anales soc. españ. fis. quim., 9, 197-8.—F was not found in H<sub>2</sub>O from the Grand Geyser and the hot springs of Reykir, but was present in H<sub>2</sub>O from the hot springs of Reykjawik. The waters of Iceland show only traces of Li.

H. S. Paine.

Classification of Clays (STREMME). 19.

Mineral Resources of the United States. U. S. Geological Survey.

Schiffner, C.: Uranmineralien in Sachsen. Freiberg: Craz & Gerlach. 20 pp., I M.

## 9. METALLURGY.

WILLIAM BRADY, WILLIAM T. HALL.

Recent Progress in Slime Filtration Development. C. S. HALEY. Mining Sci. Press, 103, 16-7.

E. J. CRANE.

Roasting of Rickel-Copper Mat. E. F. Kern and H. W. Walter. School of Mines Quart., 32, 364-88.—FeSO<sub>4</sub> oxidizes to the basic sulfate at temps. between 280° and its decomp. temp. (about 58°). The higher the temp., the more rapid the oxidation; and also, the more rapid the decomp. the higher the temp. above 58°. Basic sulfates of Cu are not formed by the direct oxidation of CuSO<sub>4</sub> below 647° (decomp. temp.), above this decomp. increases with the temp. Basic sulfates of Ni are not formed by the direct oxidation of dehydrated NiSO<sub>4</sub>, but form at temps. above its decomp. temp. (727° and 736°). Mats produced by smelting Ni-Cu ores contain the Ni as Ni<sub>2</sub>S and when "blown" to almost complete elimination of the Fe sulfide, contain an alloy of Ni and Cu in the proportion in which they existed in the mat. For rapidly roasting Ni-Cu mat, roast at 450-500° for sufficient time to coat the particles with a film of oxides, after which continue the roasting at 675 and 750° until the desired desulfurization has been accomplished. Continuous rabblings is necessary to prevent sintering. Special methods of analysis are given.

Amalgamation Experiments with the Boklevsky Centrifugal Amalgamator in Hungary. J. Grunhut. Oesterr. Z. Berg-Hattenw., 59, 363-5, 381-4, 395-7.—This app. has been described (Ibid., 57, No. 11). Expts. on low-grade Au-Ag ores, gave 30.52% Au recovery in addition to 69.48% recovered by means of the usual amalgamated Cu plates. Any mill treating free-milling ores can increase the yield considerably by the use of this app. Ores containing Cu, or anything else that makes cyaniding costly or impossible, can be utilized. The amalgam obtained needs washing only 2-3 times. The price is about \$1000 and it pays for itself in 2 yrs. E. J. Ericson.

The Application of Graphite to the Production of Crucibles for Melting Metals. I. A. HAENIG. Brass World, 7, 307-12.—The best clays for the manuf. of crucible mixts. are the Klineberger, Grosalmeroder, Grünstadter or Kärlischer varieties. The practice at the Joseph Dixon Crucible Co., Jersey City, which employs Ceylon graphite is as follows: The flake graphite is ground in a ball mill, mixed with kaolin in varying quantities, 10 parts of graphite and 7 parts of gray clay from Klingenberg and some pulverized charcoal added, the latter to secure porosity, the mass moistened with H<sub>2</sub>O

and kneaded in a vertical cylinder. Analyses of various brands of graphite crucibles are given. ROBERT KANN.

Blast Furnace Smelting with Oil. E. JACOBS. Eng. Mining J., 92, 434.—An at. of 14.6 gal. of oil per ton of material smelted was required. The rate of smelting was 110 tons per 24 hrs.

R. K.

Mining in San Juan County, Colo. W. C. PROSSER. Eng. Mining J., 92, 435-6. —The main problem there is the treatment of 2 classes of ore: (1) Cyaniding ores; (2) Zn-bearing sulfide ores. The cyaniding ores are of 3 classes and occur in as many areas.

R. K.

Cast Iron Tuyères. B. Gore. Eng. Mining J., 92, 437.

R. K.

Nomenclature of Iron and Steel. H. M. Hows. Eng. Mining J., 92, 550-1.

R. K.

Metal Resources of Philippines. W. D. SMITH. Eng. Mining J., 92, 602

R. K.

Michigan Copper Refining Practice. H. D. Conant. Eng. Mining J., 92, 494; School Mines Quart., 32, 285-7.—The present practice of using sep. furnaces for the melting and refining of Lake Superior Cu has allowed charges of over 300,000 lbs. of Cu to be poured. The diff. furnaces are described. The slags produced contain 15-30% Cu and may be acid, i. e., 40% SiO<sub>2</sub> and 20% Fe<sub>2</sub>O<sub>2</sub> if produced from amygdaloid mineral; or basic, 20% SiO<sub>2</sub> and 45% Fe<sub>2</sub>O<sub>3</sub> if produced from conglomerate.

R. K.

Potential Mineral Resources of Maine. Anon. Eng. Mining J., 92, 595.

R. K

The Wislicenus Smokestack. O. H. Hahn. Eng. Mining J., 92, 596-7.—A method of stack construction which results in a greatly accelerated mixing of the waste gases with air. The sides of the stack contain a great number of openings. R. K.

Auriferous Gravels of Cariboo. B. C. E. JACOBS. Eng. Mining J., 92, 598-602.—Profitable quantities of placer Au are assured.

R. K.

The Conservation of our Metal Resources. A. E. Greene. Chicago, Ill. J. Ind. Eng. Chem., 3, 674-7.—An address.

J. J. M.

The Iron and Steel Industries of Sheffield. J. O. ARNOLD AND A. McWILLIAM. J. Soc. Chem. Ind., 30, 995-7.

J. J. M.

The Silver Industry of Sheffield. E. A. SMITH. J. Soc. Chem. Ind., 30, 997-9.

J. J. M.

Future Economies in Rand Reduction Plants. C. O. Schmitt. J. Chem. Met. Soc. S. Africa, 12, 16-30.

J. J. M.

Lining Alloys. H. Greenburg. Met. Chem. Eng., 9, 473. J. J. M.

Primitive Metal Coloring. O. H. Evans. Metal Ind., 9, 346-7. J. J. M.

The Coloring and Staining of Metals. T. HADDOW. Metal Ind., 9, 382.

J. J. M.

Mining and Metallurgical Treatment of the Gold Ores of Colorado. E. ACKER-MANN. Rev. chim. ind., 22, 251-5. J. M.

Generalities on Aluminium. A. GAUTHIER. Rev. chim. ind., 22, 255-8.

J. J. M.

Silicon Steel. G. A. BISSET. Iron Age, 86, 442-5.—By adding Si to low-C steel it is possible to obtain as high a tensil strength as that obtained with high-C steel, while other valuable characteristics of a low-C steel are maintained. The ductility may be less but the reduction of area and the impact resistance are higher. Illusts.

and tables are given showing the results of tests on destruction of flasks for compressed air, made of low-C, high-Si and V-Cr-Ni and V-Cr steels. Strength combined with lightness and non-shattering on failure were the points desired. The flasks made of V-Cr steel were vastly superior in every way; but the Si steel used should have had a C content of 0.20-0.24% instead of 0.39%. Other tables are given showing the results of various comparative tests on rivets, rods and plates of Si and ordinary steel.

L. A. TOUZALIN.

The Continuous System of Cyaniding in Pachuca Tanks. HUNTINGTON ADAMS. Bull. Am. Inst. Min. Eng., 56, 595-601.—An arrangement permitting of continuous agitation allows no part of the pulp to pass out of the tank, without having gravitated to the bottom, and risen through an air lift tube, thoroughly mixed. The advantages were found to be simplicity in installation, accessibility for handling, no plugging of pipe connections, and no classification of slimes.

E. WALLER.

The Preparation of Brown Iron Ores. H. S. Geismer. Bull. Am. Inst. Min. Eng., 56, 643-53.

Amalgamation of Gold in Banket Ore. W. R. Dowling. J. Chem. Met. Soc. S. Africa, 11, 609-14.—Discussion of C. A., 5, 2241.

E. WALLER.

The Behavior of Sulfur in Cupola Furnace Fusions. C. PARDUN. Stahl u. Eisen, 31, 665-q.—The question was studied by the author, by running several heats, and drawing off small samples of the iron (4 or 5 for each heat) at frequent intervals, the samples being afterward analyzed for S. One heat was run with fore-hearth without any addition; 2 and 3 using limestone and slag resp. with fore-hearth; 4 and 5 using same without fore-hearth; in 6 the hot blast was driven through the coke charge for some minutes before the Fe was charged in. The coke used contained 1% S. The conclusions are: (1) Much of the S in the coke passes into the Fe first used. (2) According to Wüst, the S from the SO, gases passes into the glowing Fe before it is fused. Covering the metal with a protective coating of lime does not hinder this action. (3) It is not possible to obviate the absorption of S by use of limestone or of slag. (4) The action of such additions only occurs after a certain period when the bath and the slag are together in sufficient quantity and are hot enough. Desulfurizing then occurs, because the S compds. dissolved in the Fe rize to the top and are taken up by the slag. (5) The most decided desulfurization occurs in the crucible and it is materially aided by agitation or stirring (Simmersbach). (6) Though Mn plays an important part in desulfurizing, considerable additions of Mn are not advizable, as a large proportion remains in the Fe. Metal No. 6 reduced the high S content of the first runnings, but left much to be taken out in the crucible. E. WALLER.

The Strength of Steels in Compound Stress, and Endurance under Repetition of Stress. L. B. TURNER. Engineering, 92, 305-9.

J. J. M.

Wastes of a Blast Furnace (HAGAR). 20.

AUSTIN, L.: The Metallurgy of the Common Metals, Gold, Silver, Iron, Copper, Lead and Zinc. 3rd. Ed. San Francisco: Mining and Scientific Press. 8°, 528 pp., \$4.00.

Breull, P.: La metallurgie a l'exposition universelle et internationale de Bruxelles de 1910. Paris: Dunod & Pinat. 4°, 15 Fr.

DIEGEL, C.: Einige Versuche mit der autogenen Schweissung von Flusseisen. Berlin: L. Simion. 2.50 M.

FRIEND, J. N.: The Corrosion of Iron and Steel. London: Longmans & Co.  $8^{\circ}$ , 6 s.

KRANTZ, F.: Die Entwicklung der oberschlesischen Zinkindustrie in technischer, wirtschaftlicher und gesundheitlicher Hinsicht. Kattowitz: Böhm. 8°, 92 pp., 6 M.

KRAUSS, A.: Eisenhüttenkunde. Leipzig: Goschensche Verlagshandlung. 2 Bände, je 80 Pf.

Brit., 20,989, Sept. 8, 1910. R. J. McNitt. Separation of Alkali metals from alloys; see C. A., 5, 2484.

Ger., 236,244, Jan. 15, 1910. ART.-GES. MIX & GENEST TELEPHON- U. TELE-GRAPHEN-WERKE, Schöneberg bei Berlin. In the pretreatment of aluminium and alloys for galvanizing, the purified and cleaned Al is dipped into a heated bath composed of acids of the halogen group with the addition of reducing agents such as alc., which retard the otherwise very violent reaction. After a short time the Al is removed from the bath and galvanized in the usual manner in the known baths. The resulting coatings are very permanent and admit well of polishing.

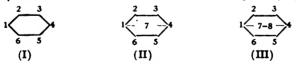
#### 10. ORGANIC CHEMISTRY.

I. BISHOP TINGLE.

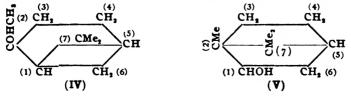
Albert Ladenburg. W. HERZ. Chem. Ztg., 35, 933-4.—Obituary. J. J. M.

Homologs of Tyrosine. J. ALOY AND C. RABAUT. Chem. Ztg., 34, 931; J. pharm. chim., 3, 481-3.—Lower homologs of tyrosine were prepared by treating anisaldehyde with KCN and NH<sub>4</sub>Cl; the aminonitrile obtained on being hydrolyzed by dil. HCl gave p-methoxyphenylaminoacetic acid which on demethylating with conc. HI yields p-hydroxyphenylaminoacetic acid, HOC<sub>6</sub>H<sub>4</sub>CH(NH<sub>2</sub>)CO<sub>2</sub>H, needles. Cu salt; hydrochloride; hydrobromide; hydrodide cryst.; it gives Millon and Pircas' reactions and differs from tyrosine in its action on hypochlorites. The higher homolog "ratanhia," C<sub>6</sub>H<sub>12</sub>NO, could not be found in ratanhia but it occurs in various species of Andira (A. spectabilis, A. enerinis).

Nomenclature of Polycyclic Hydrocarbon Chains. GRIGNARD. Chem. Zig., 34, 941.—Baeyer's nomenclature of cyclic compds. with inner chains often presents difficulties in naming certain compds. and in no case does it give a name in which the character of the inner chain is clear at a glance. The author proposes a modification in nomenclature, which avoids confusion. The names of compds. (I), (II) and (III)



are according to Baeyer bicyclo-0,2,2-hexane, bicyclo-1,2,2-heptane and bicyclo-2,2,2-octane, resp.; according to the new method bicyclo-1,4-hexane, bicyclo-1,7,4-hexane and bicyclo-1,7,8,4-hexane, resp. If there is a double linkage in the bridge the prefix becomes "bicycleno" and its position is shown by an accent between the 2 position nos., thus 7<sup>1</sup>; 8. Pinene hydrate (IV) can be changed into borneol (V). Ac-



cording to Baeyer (IV) is trimethyl-2,7,7-bicyclo-1,1,5-heptanol-2, and (V) trimethyl-1,7,7-bicyclo-1,2,2-heptanol-1; according to the author they are trimethyl-2,7,7-bicyclo-1,7,5-hexanol-2 and trimethyl-2,7,7-bicyclo-2,7,5-hexanol-1, resp.

E. J. WITZEMANN.

Action of Colloidal Metals (Platinum, Gold, Silver and Palladium) Prepared by Bredig's Method on Solutions of Guaiaconic Acid. G. A. BUCKMASTER. 7th Intern. Congr. Applied Chem., 1909 (Sect. IV, A 2), 29; J. Chem. Soc., 100, I, 390. J. J. M.

Constitution of Scopoletin. C. W. Moore. Wellcome Chem. Research Lab. Proc. Chem. Soc., 27, 119; J. Chem. Soc., 99, 1043-8.—Scopoletin, a fluorescent compd. obtained from Scopolia japonica and other plants, is 4-hydroxy-5-methoxycoumarin, since by prolonged b. with KOH, 2,4-dihydroxy-5-methoxycinnamic acid, C<sub>0</sub>H<sub>1</sub>O<sub>2</sub>OMe, H<sub>2</sub>O, results; bright yellow needles from H<sub>2</sub>O, m. 178-80° (decomp.). The following compds. have been prepared: 2,4-Diacetoxyanisole, C<sub>10</sub>H<sub>0</sub>O<sub>4</sub>OMe, prisms from MeOH, m. 62-4°. 2,5-Diacetoxyanisole, prismatic needles from MeOH, m. 93-4°. 2,4,5-Trimethoxycinnamic acid, C<sub>10</sub>H<sub>12</sub>O<sub>4</sub>, m. 163-5°. 2,4-Dihydroxy-5-methoxy-β-phenyl-propionic acid, C<sub>10</sub>H<sub>12</sub>O<sub>4</sub>, H<sub>2</sub>O, needles from H<sub>2</sub>O, m. 155°. 2-Hydroxy-4-acetoxy-5-methoxy-β-phenyl-propionic acid lactone, C<sub>11</sub>H<sub>12</sub>O<sub>4</sub>, plates from MeOH, m. 135°. 2,4,5-Trimethoxy-β-phenyl-propionic acid, C<sub>12</sub>H<sub>12</sub>O<sub>4</sub>, needles with 1 mol. H<sub>2</sub>O, m. 74-76°; anhyd. m. 95-6°. Methyl ester, needles from MeOH, m. 54°.

Mannitoboric Acid. J. J. Fox and A. J. H. Gauge. E. London Coll. *Proc. Chem. Soc.*, 27, 136; *J. Chem. Soc.*, 99, 1075–9.—Mannitoboric acid, C<sub>2</sub>H<sub>15</sub>O<sub>2</sub>B, prisms, m. 89.5°, is prepared from mixts. of mannitol and H<sub>2</sub>BO<sub>2</sub> dissolved in abs. EtOH. It is a monobasic acid yielding normal ammonium, silver, calcium and barium salts. These are pptd. from aq. solns. by EtOH. Measurements of the rotation of mixtures of mannitol and H<sub>2</sub>BO<sub>2</sub> in abs. alc. indicate that the formation of mannitoboric acid is favored by increasing the amt. of either compd.

E. E. Gorsline.

Course of Chemical Change in Quinol under the Influence of Radiant Energy. W. N. HARTLEY AND O. H. LITTLE. Royal Coll. of Science, Dublin. Proc. Chem. Soc., 27, 137; J. Chem. Soc., 99, 1079–91.—Over 36 expts. were made on quinol in a state of vapor, in darkness, diffused daylight, and in ultraviolet light, flasks of glass and of quartz being used. The course of chem. change was measured by the amt. of H evolved. The reaction  $C_xH_4(OH)_2 \longrightarrow C_0H_4O_2 + H_2$  seems to attain equil. when 0.6 mg. mol. of quinol has lost 6.7 cc. of H, and the principal product is quinhydrone. The reaction is not strictly reversible in the presence of air or of O, as the quinhydrone does not revert to quinone except in an atm. of H. In a flask containing cryst. quinol, quinhydrone and p-benzoquinone, exposed to daylight, the p-benzoquinone soon disappears, its vapor reacting with the residual quinol to form quinhydrone. The most complete and rapid chemical change was effected in every case, by rays of the shortest wave length.

Intramolecular Condensation of Aromatic Sulfinic Acids. II. The Interaction of Aromatic Disulfoxides and Sulfuric Acid. T. P. HILDITCH. Univ. London. *Proc. Chem. Soc.*, 27, 139; *J. Chem. Soc.*, 99, 1091-1100.—The decomp. of aromatic disulfoxides by cold conc. H<sub>3</sub>SO<sub>4</sub> is dependent chiefly on the number and position of the alkyl substituents in the aromatic nucleus. In all cases the primary reaction can only be explained by supposing that the disulfoxide mol. undergoes hydrolytic fission

with formation of a mixt. of sulfinic and (unstable) sulfoxylic acids, RSOSOR RSO<sub>2</sub>H + RSOH. The intermediate products may in certain cases unite with more

complex derivs. such as oxides of the diphenylene disulfides, or in the presence of a phenolic ester, many condense to simple or complex sulfonium bases. Diphenyl disulfoxide in cold conc. H<sub>2</sub>SO<sub>4</sub> yields diphenylene o-disulfoxide monoxide and the corresponding p-disulfide. When phenetole is added in the above reaction there is found, thiodiphenylenephenetylsulfonium chloroplatinate, C40H40,S4Cl4Pt, brown powder, m. 191°. and phenylthiolphenyldiphenetylsulfonium chloroplatinate, [(PhSCaH4)(EtOCaH4)a-SCI , PtCl, brown cryst., m. 130°. These 2 compds. are obtained by dissolving the mixed chloroplatinates in epichlorohydrin from which soln. the former is pptd. by alc. Diphenylene-o- and b-disulfide monoxides were each dissolved in cold conc. H<sub>2</sub>SO<sub>4</sub> and phenetole added. In neither case could a sulfonium base be detected. By suspending p-phenetylsulfinic acid in very dil. H,SO4, warming and adding a few drops of HI together with sufficient H<sub>2</sub>SO<sub>2</sub> to decolorize the soln., di-p-phenetyldisulfoxide, C10H18O4S2, results; yellow tablets, m. 89°. When this compd. is condensed with phenetole, di-p-phenetylsulfone and triphenetylsulfonium chloroplatinate are obtained. Triphenetylsulfonium dichromate, C41H44O11S1Cr2, orange cryst., m. about 65°. Di-ptolyl methyl ether disulfoxide, C10H18O4S2, was obtained by sulfonating p-tolyl methyl ether in the presence of AlCl, using petroleum ether, b. 95-7°, as a solvent; the resulting sulfinic acid was converted by the HI acid method; yellow prisms, m. 83-6°. By reducing this sulfoxide by HI and glac. AcOH there results di-p-tolyl methyl ether disulfide, C1eH12O2S2, tablets, m. 73-4°. The disulfoxide with H2SO4 gives a compd. MeOC, HasC, Ha (OMe) (SO) a C, Ha (OMe) SOC, Ha OMe, soft laminae, m. 184°. The addition of p-tolyl methyl ether to the soln. of the disulfoxide in H<sub>2</sub>SO<sub>4</sub> produced no change, the above product being isolated. E. E. GORSLINE.

Hydroaromatic Ketones. II. 1,1,2-Trimethylcyclohexan-3-one. A. W. Crossley and N. Renouf. Pharmaceutical Soc. Proc. Chem. Soc., 27, 137; J. Chem. Soc., 99, 1101-12.—When 1,1,2-trimethyldihydroresorcinol (J. Chem. Soc., 79, 141 (1901)) is treated with PCl<sub>4</sub> there results, chlorotrimethylcyclohexenone, C<sub>4</sub>H<sub>12</sub>OCl, a refractive liquid, b<sub>43</sub> 130°. Semicarbazone, plates, m. 185-6° (decomp.). Oxime, needles, m. 121°. When oxidized by KMnO<sub>4</sub> δ-acetyl-δ-methylhexoic acid and α,α-dimethyladipic acid result. When chlorotrimethylcyclohexenone is distilled trimethyldihydroresorcinol anhydride, C<sub>18</sub>H<sub>28</sub>O<sub>2</sub>, is recovered from the residue; needles, m. 158-9°. 1,1,2-Trimethylcyclohexane-3-ol. (I) is obtained by reducing chlorotrimethylcyclo-

hexenone with Na in moist Et<sub>2</sub>O; it possesses a camphoraceous odor, is extremely sol. in organic solvents and m. 28°. Benzoyl derivative, needles, m. 61°. m-Nitrobenzoyl derivative, scaly needles, m. 44°. o-Nitrobenzoyl deriv., yellow needles, m. 114°. When oxidized with KMnO<sub>4</sub>, δ-acetyl-δ-methylhexoic acid results. When Beckmann's CrO<sub>3</sub> mixture is employed for the oxidation 1,1,2-trimethylcyclohexan-3-one, (II) results; refractive liquid, b<sub>780</sub> 190.5-1°. Semicarbazone, scales, m. 206° with gas evolution. Oxime, needles, m. 95°, possesses a sickly camphoraceous odor. Oxidation of this ketone yields δ-acetyl-δ-methylhexoic acid, MeCOC(Me)<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>CO<sub>2</sub>H, viscid liquid, b<sub>26</sub> 188.5°. Semicarbazone, needles, m. 169° with gas evolution. Silver salt, needles, m. 104-5°. Oxidation of this acid yields α,α-dimethyladipic acid. E. E. G.

Electrolytic Reduction. IV. Aromatic Aldehydes. H. D. LAW. Borough Polytech. Inst. Proc. Chem. Soc., 27, 138; J. Chem. Soc., 99, 1113-7.—Previous work (C. A., 1, 173) has shown that aromatic aldehydes, when reduced by the electrolytic method, yielded resinous products in varying amts. which is least when the groups around the C<sub>2</sub>H<sub>2</sub> nucleus occupy asymmetrical positions. The amt. increases as the

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deviation from this structure becomes greater. An exam. of the 3 chlorobenzaldehydes confirms these results. When a nearly neutral medium is used for the reduction the amt. of resinous products formed is greatly reduced.

E. E. GORSLINE.

Synthetical Experiments in the Group of the iso-Quinoline Alkaloids. I. Anhydro-cotarninephthalide. E. HOPE AND R. ROBINSON. Univ. Manchester. Proc. Chem. Soc., 27, 125; J. Chem. Soc., 29, 1153-69.— Anhydrocotarnine-5-nitrophthalide (I),

is made by dissolving 20 g. cotarnine in 100 cc. of EtOH and gradually adding 16 g. of 5-nitrophthalide. After b. for 10 min. the product is washed with EtOH; pale yellow cryst. m. 176-7° (decomp.). Picrate, yellow prisms, m. 159-61°. Chloroplatinate, yellow, amorphous. Chloroaurate, buff. Mercurichloride, colorless cryst. This compd. is reduced by dissolving in glac. AcOH and quickly adding Sn and hydrated SnCl<sub>2</sub> dissolved in conc. HCl, at 15-8.° Anhydrocotarnine-5-aminophthalide, C<sub>20</sub>H<sub>20</sub>-O.N., prisms, m. 220-1°. Sparingly sol. in C.H., Et.O or McOH. Chloroplatinate, Picrate, bright yellow. Anhydrocotarnine-5-acetylaminophthalide, pale yellow. C<sub>20</sub>H<sub>22</sub>O<sub>4</sub>N<sub>2</sub>, CH<sub>4</sub>O, results from shaking the amino compd. with enough Ac<sub>2</sub>O to dissolve it; prisms, m. 247° (decomp.). By treating the amino compd. dissolved in conc. HCl with NaNO, and then pouring the mixture in a dil. soln. of KI, anhydrocotarnine-5-iodophthalide, C20H18O2NI, results; hexagonal plates from MeOH, m. 173°. The hydrochloride and sulfate of this base are sparingly sol. in cold H<sub>2</sub>O. Chloroplatinate, colorless amorphous. Chloroaurate, pale yellow. Mercurichloride, white, amorphous. By diazotizing the amino compd., anhydrocotarnine-5-hydrazinophthalide, CH<sub>2</sub>: O<sub>2</sub>: CaH(OMe): CaHaHCaOa : CaHaNHNHa is formed; colorless needles from MeOH, decomp. 113-6°. Benzoyl derivative, pale yellow prisms from MeOH, m. 230-2°. Picrate, bright yellow. Anhydrocotarninephthalide, CH, : O, : CaH(OMe) : CaHaNCaHO2 : CaHa, is formed: (a) by direct condensation of cotarnine with phthalide; (b) by the condensation of cotarnine with nitrosophthalimidine; (c) by the oxidation of anhydrocotarnine-5-hydrazinophthalide; long hexagonal prisms from MeOH or Et<sub>2</sub>O, m. 201-2°. Chloroplatinate, nearly colorless, insol., amorphous, Mercurichloride. Chloroaurate. Picrate, yellow prisms, m. 202-3°. In method (b) there was obtained a picrate, octahedron, m. 240-1.° It is isomeric with the other and probably derived from a compd. of the type of nornarceine. When anhydrocotarninephthalide is oxidized by HNO<sub>21</sub> or by MnO<sub>2</sub> and dil. H<sub>2</sub>SO<sub>4</sub>, the products are cotarnine and phthalaldehydic acid. By b. anhydrocotarninephthalide with MeI, the methiodide results; prisms, m. 207° (decomp.). Upon treating with a slight excess of NaOH, collecting the pptd. methylhydroxide, and b. with a large excess of H<sub>2</sub>O, this compd. yields dedimethoxynarceine, (II), lustrous laminae, m. 189-90°. In its reactions this compd. shows great analogy to narceine. Hydrochloride, needles, m. 172-4°, with gas evol. Chloroplatinate, yellow needles. E. E. GORSLINE.

Synthesis of Pinacols (Pinacones). I. W. PARRY. Merchant Taylors' School,

Liverpool. Proc. Chem. Soc., 27, 141; J. Chem. Soc., 99, 1169–74.—By using the appropriate Grignard reaction on Et  $\alpha$ -hydroxyisobutyrate (C. A., 4, 3216) the following pinacols (pinacones) have been prepared: A pinacol isolated as its hydrate, HOCMe<sub>2</sub>CMe<sub>2</sub>OH.6H<sub>2</sub>O, m. 44–5°.  $\beta$ -Methyl- $\gamma$ -ethyl-pentane- $\beta$ , $\gamma$ -diol, viscid oil, b<sub>20</sub> 100°. Repeated fractionation under atm. pressure eliminates H<sub>2</sub>O, giving diethyl-isopropenylcarbinol, CH<sub>2</sub>: CMeCEt<sub>2</sub>OH, b. 150–2° and having the odor of camphor.  $\beta$ -Methyl- $\gamma$ -propylhexane- $\beta$ , $\gamma$ -diol, HOCMe<sub>2</sub>CPr<sub>2</sub>OH, viscid oil, b<sub>18</sub> 105°.  $\alpha$ ,  $\alpha$ -Diphenyl- $\beta$ -methyl-propane- $\alpha$ , $\beta$ -diol, HOCMe<sub>2</sub>CPh<sub>2</sub>OH, silky needles, m. 89–9.5°. It is dehydrated by Ac<sub>2</sub>O containing a few drops of H<sub>2</sub>SO<sub>4</sub> and a compd. which is probably d<sub>1</sub>l-diphenyl-

CMe<sub>3</sub>
dimethylethylene oxide, | O, results; prisms, m. 44-5°. α,α-Dibenzyl-β-methyl-CPh<sub>2</sub>

propane- $\alpha,\beta$ -diol, HOCMe<sub>2</sub>C(CH<sub>2</sub>Ph)<sub>2</sub>OH, prisms, m. 47-8°. At the same time a small quant. of an unsatd. alc. is formed, b<sub>12</sub> 165-8°. This is probably dibensylisopropenyl-carbinol, CH<sub>2</sub>: CMeC(CH<sub>2</sub>Ph)<sub>2</sub>OH.  $\alpha,\alpha$ -Dinaphthyl- $\beta$ -methylpropane- $\alpha,\beta$ -diol, HOCMe<sub>2</sub>-C(C<sub>10</sub>H<sub>7</sub>)<sub>2</sub>OH, m. 166-6.5°. The solid pinacols have normal mol. wts. in dil. C<sub>2</sub>H<sub>4</sub>. E. E. GORSLINE.

Decomposition of Diethylenesulfidemethylsulfine Hydroxide in Aqueous Solution. L. Green and B. Sutherland. Univ. Melbourne. Proc. Chem. Soc., 27, 140; J. Chem. Soc., 99, 1174-8.—The hydroxide, corresponding with the salts of the sulfine, decomps. spontaneously into  $H_2O$  and a neutral oil (Masson, Proc. Chem. Soc., 49, 223 (1886); Mansfield, Ber., 19, 696, 2658 (1886); V. Meyer, Ibid., 3262). The velocity of the reaction has been measured at 55° in solns. sufficiently dil. to assume total ionization. Solns. of the pure hydroxide and others, to which was added NaOH or  $C_3H_{11}S_3I$ , were used. The action is of the second order. The velocity is directly proportional to the conc. of each of the ions of the sulfine base. It may be represented by the equation:  $C_3H_{11}S_3 + OH' = C_2H_{10}S_2 + H_2O$ .

Purification of Acetic Acid. K. J. P. Orton, M. G. Edwards and H. King. Univ. Coll. of N. Wales. Proc. Chem. Soc., 27, 120; J. Chem. Soc., 99, 1178-81.—Cl and Br do not react with pure AcOH when light is excluded. A large part of com. AcOH contains impurities which react with these elements. To remove these the acid which must not m. below 16° is distilled with P<sub>4</sub>O<sub>10</sub> using a quant. of the latter necessary to form H<sub>2</sub>PO<sub>4</sub> with the H<sub>2</sub>O present. Using 2 l. of the acid the first 50 cc. of distillate is discarded. 1700 cc. of AcOH can be collected, m. 16.4-16.5°, free from impurities combining with Cl and Br, and containing only 0.009-0.04 g. of Ac<sub>2</sub>O per 100 cc. of the acid. The acid does not seem to deteriorate on standing without special precaution to exclude light and air.

Detection and Estimation of Small Quantities of Acetic Anhydride in Acetic Acid. M. G. Edwards and J. K. P. Orton. Proc. Chem. Soc., 27, 121; J. Chem. Soc., 99, 1181-5.—2,4-Dichloroaniline reacts with Ac<sub>2</sub>O with great rapidity. AcOH is treated with this compd. and after a few hrs. at 16° the mixture is extracted with CHCl<sub>2</sub>. The dichloroaniline is separated from the anilide by shaking with 10% HCl and the anilide indirectly estimated as chloroamine.

E. E. GORSLINE.

Method of Chlorination. Chlorination of Anilines and Phenols. K. J. P. ORTON AND H. KING. Proc. Chem. Soc., 27, 139; J. Chem. Soc., 99, 1185-92.—The method of chlorination is based on the reversible interaction between chloroamine and HCl, (Orton and Jones, C. A., 3, 2973). By use of the proper amt. of HCl any conc. of Cl can be obtained. This conc. will remain constant during the chlorination because a mol. of Cl used in chlorination will be replaced by a mol. of HCl, which in its turn reacts with a further amt. of chloroamide: ArNClAc + HCl = ArNHAc + Cl<sub>3</sub>. In this way oxidation and side reactions are avoided and the operations effectively con-

trolled. The usual procedure is to mix equiv. quants, of the compd. to be chlorinated and the chloroamide in glac. AcOH and then and  $\frac{1}{100} - \frac{1}{20}$  g. mol. of HCl in the form of a const. b. aq. soln. (22 g. HCl per 100 cc.). The mixture is kept in the dark at 1.5° and at intervals 1 cc. is withdrawn and the remaining chloroamine and Cl estimated to det. when the reaction is complete. The time required varies from a few mins. to 2 or 3 ds. The chloroamines most frequently used are 2,4-dichloroacetanilide and p-nitroacetanilide. The following compds. were prepared. 5-Chloroaceto-mxylidide, C10H110NCl, prisms, m. 196.5-7.5°. 5-Chloro-m-xylidine, C2H10NCl, feathery needles, m. 37-8°. 6-Chloroaceto-F-cumidide, C11H14ONCl, needles, m. 208-9°. 6-Chloro-F-cumidine, C.H., NCl, needles, m. 55-6°. 5-Chloro-o-anisidine, C.H., ONCl, flat needles, in. 45-6°. 5-Chloroaceto-o-phenetidide, C10H110,NCl, six-sided plates, m. 105.5-6.5°. 5-Chloro-o-phenetidine, C<sub>2</sub>H<sub>10</sub>ONCl, flat needles, m. 21-2°. 5-Chloroaceto-p-phenetidide, needles, m. 128-9°. 5-Chloro-p-phenetidine, plates or scales, m. 64-5°. 5-Chloro-m-xylenol, b. 221-3°. 2,4-Dichloroacetanilide has a solubility of 6.4 g. per 100 cc. in glac. AcOH and 0.83 per 100 cc. in 50% AcOH. p-Nitroacetanilide has a solubility of 0.83 g. per 100 cc. in glac. AcOH and 0.38 g. per 100 cc. in 50% AcOH. E. E. GORSLINE.

Some Reactions of &-Bromomethylfurfuraldehyde. W. F. Cooper and W. H. NUTTALL. Cooper Lab., Watford. Proc. Chem. Soc., 27, 134; J. Chem. Soc., 99, 1103-1200.—w-iso-Cyano- or w-cyanomethylfurfural cannot be prepared by the action of KNC or AgNC on the bromo compd. in alc., since the great reactivity of Br in the ω-position causes a condensation between the alc. and aldehyde. ω-Ethoxymethylfurfural results and HBr is liberated, which reacts with AgNC causing a liberation of HNC. The same ethoxyaldehyde results from b. an alc. soln. of the bromo aldehyde with excess of CaCO<sub>2</sub>. w-Ethoxyfurjural, C<sub>8</sub>H<sub>10</sub>O<sub>2</sub>, is a yellow oil, d<sub>18.6</sub> 1.1096, b. 235-40°. Phenylhydrazone, yellow needles, m. 55.5-6.2°. p-Bromophenylhydrazone, silky needles, m. 104-5°. By oxidizing the above aldehyde with Ag<sub>4</sub>O there is formed, w-ethoxymethylpyromucic acid, C<sub>2</sub>H<sub>10</sub>O<sub>4</sub>, colorless needles, m. 57.5-8.5°. Silver salt. w-Methoxymethylpyromucic acid, CaHaOaOMe, was prepared by oxidizing the aldehyde obtained from MeOH and bromomethylfurfural with Ag<sub>2</sub>O; silken needles, m. 67.5-8.5°. When ω-bromomethylfurfural is b. in H<sub>2</sub>O in the presence of BaCO, it gives w-hydroxymethylfurfural; under certain conditions, however, a yellow cryst. compound results, C,1H,6O4, m. 111.5-12°. E. E. GORSLINE.

Some Derivatives of Gelsemine. C. W. Moore. Wellcome Chem. Research Lab. Proc. Chem. Soc., 27, 157; J. Chem. Soc., 99, 1231-40.—Gelsemine, C. H. O. N. (C. A., 4, 763), is stable toward alkali hydroxides. The mol. is decompd. by oxidizing agents but no definit compds. have been isolated. On b. with conc. HCl for 3 hrs. 3 new bases are found: The chief product, apogelsemine, C20H21O2N2, cannot be cryst. Hydrochloride, C20H24O3N2.HCl.H2O, glistening needles, m. 250-60°; after drying, m. 115-20°. Methiodide, prisms, m. 295° (decomp.). Diacetylapogelsemine hydrochloride, C, H, ON, (OAc), HCl, plates, m. 286°; [a] 21.7°. Monoacetylapogelsemine, C2H2O,N2, prisms, 295-8°. Some chloroisoapogelsemine, C20H2O2N2CI, was also obtained; prisms, decomp. about 220°. Chloroaurate, C. H. O. N. Cl. HAuCl. H<sub>2</sub>O, orange prisms, decomp. 160°. Methiodide, prisms, m. 265° (decomp.). Chloroacetylisoa pogelsemine, CaoHaoONaClOAc, prisms, m. 180°. When chloroisoa pogelsemine is b. with EtaNPh it loses the elements of HCl giving a base apparently isomeric with gelsemine; m. 140-5°, [a] 25.2°. The third product is isoapogelsemine, Co-HaO.N., prisms, m. about 310°. It is insol. in most organic solvents except pyridine; [α]<sub>D</sub> 16.6. Hydrochloride, C<sub>20</sub>H<sub>24</sub>O<sub>2</sub>N<sub>2</sub>.HCl.H<sub>2</sub>O, [α]<sub>D</sub> 27.1°. Methiodide, plates, m. 266° (decomp.). [α] 28.1°. Diacetylisoapogelsemine hydrochloride, C<sub>20</sub>H<sub>20</sub>ON<sub>2</sub>-(OAc), HCl.H<sub>2</sub>O, plates, m. 305°; [a] 24.6°. Bromoisoapogelsemine, C.H., O, N, Br, is obtained together with apo- and isoapogelsemine by b. gelsemine with HBr; plates which decomp. at about 220°.

E. E. GORSLINE.

Constituents of the Bulb of Buphane disticha. F. TUTIN. Wellcome Chem. Research Lab. Proc. Chem. Soc., 27, 149; J. Chem. Soc., 99, 1240-8.—Buphane disticho is a bulbous plant, a native of South Africa where it is known as the "poison bulb." The inner portions of the bulb contain a large amt. of alkaloid, while the dry, outer portions are free from this substance. An alc. extract of the inner portions of the bulb yield a small amt. of an essential oil containing furfuraldehyde. The nonvolatil product which is sol. in H<sub>2</sub>O, yielded acetovanillone, chelidonic acid and considerable amts. of levulose and Cu. A mixture of alkaloids was also present, the principal constituent being buphanine, of which no cryst, deriv, could be prepared. It possesses a physiological action similar to hyoscine. When heated with a large excess of KOH, a CHCl<sub>2</sub> extraction yields buphanitine, C<sub>21</sub>H<sub>24</sub>O<sub>6</sub>N<sub>22</sub> colorless prisms from EtOH, which loses solvate at 130° and m. 240°. It is readily sol. in CHCl, and AcOEt and sparingly sol. in cold EtOH and H<sub>2</sub>O. Hydrochloride, C<sub>22</sub>H<sub>24</sub>O<sub>2</sub>N<sub>2</sub>.HCl, needles, m. 265-8° (decomp.). Methiodide, prisms, m. 278° (decomp.). A weakly basic alkaloid, one sol. in H<sub>2</sub>O and a small amt. of narcissine were also obtained. The first is a convulsant poison, while that sol. in H<sub>2</sub>O resembles colchicine and narcissine in its action. The portion of the original extract which was insol. in H<sub>2</sub>O yielded amorphous products, together with pentatriacontane, a phytosterol, ipuranol, and a mixt. of aliphatic acids, free and combined. E. E. GORSLINE.

Absorption Spectra of Cinchonine, Quinine and their Isomerides. J. J. DOBBIE AND A. LAUDER. Gov. Lab., London, and Edinburgh and E. of Scotland Coll. of Agr. Proc. Chem. Soc., 27, 148; J. Chem. Soc., 99, 1254-61.—Each alkaloid gives 2 distinct spectra, A and B. The band in B is more sharply defined and persistent than the band in A. The spectrum A is obtained when the alkaloid is an alc. or H<sub>2</sub>O, containing not more than 1 equiv. of acid to 1 of base; B, when it is dissolved in H<sub>2</sub>O soln. containing at least 2 equivs. of acid to 1 of base. The difference in the 2 spectra is associated with the fact that the N of the quinoline nucleus is tervalent in the former, but quinquivalent in the latter case. The spectrum A of cinchonine is practically identical with the spectrum of quinoline in alc., and B with the spectrum of quinoline salts in aq. soln. The absorption spectrum of the isomerides examined are indistinguishable from those of cinchonine and quinine resp.

E. E. GORSLINE.

Influence of Conjugated Linkings on General Absorptive Power. II. Some Openchain and Cyclic Compounds. C. R. GRYMBLE, A. W. STEWART, R. WRIGHT AND F. W. REA. Sir Donald Currie Lab. and Queen's Univ., Belfast. Proc. Chem. Soc., 27, 153; J. Chem. Soc., 99, 1262-70.—Continuing the previous work (C. A., 5, 2850). the absorption spectra of isomeric compds. containing double bonds in different positions in the mol. have been examined. In 18 cases it has been found that conjugation of double linkings increases the general absorptive power of the compd. In the case of crotonic and allylacetic acids, although the former compd. contains fewer ats. and has the same number of double bonds, yet its absorption power is very much more marked than the latter, in which the double bonds are not conjugated with each other.

E. E. GORSLINE.

New Derivatives of Aminolauronic Acid. J. Weir. Univ. St. Andrews, Univ. Cambridge. Proc. Chem. Soc., 27, 154; J. Chem. Soc., 99, 1270-7.—By dissolving 1 mol. of aminolauronic acid hydrochloride and 1 mol. of chloroacetic acid in 3 mols. of 10% NaOH and b. for 5 hrs. there is obtained carboxymethylaminolauronic acid, CO<sub>2</sub>HC<sub>2</sub>H<sub>14</sub>NHCH<sub>2</sub>CO<sub>2</sub>H, m. 183-4°; [a]<sub>2</sub><sup>18°</sup> 45.7° in H<sub>2</sub>O. On fusing this acid, N-anhydrocarboxymethylaminolauronic acid, (I), results; m. 142-3°; [a]<sub>2</sub><sup>18°</sup> —22.2° in H<sub>2</sub>O, —11.6 in EtOH, —22.2° in C<sub>2</sub>H<sub>3</sub>. By heating the Ba salt of this acid to 300°.

anhydromethylaminolauronic acid, C<sub>10</sub>H<sub>17</sub>ON, an orange-colored wax, results. I mol. aminolauronic acid hydrochloride, I mol. BzCl and 3 mols. 10% NaOH, upon shaking,

yield benzoylaminolauronic acid, CO<sub>2</sub>HC<sub>2</sub>H<sub>14</sub>NHBz, prisms, m. 204°;  $[\alpha]_{\rm p}^{190}$ —18.4° in EtOH. Methylbenzoylaminolauronate, m. 88–9°;  $[\alpha]_{\rm p}^{19}$ —28.9° in EtOH. Benzoylaminolauronic acid AcCl and PCl<sub>2</sub> yielded N-anhydrobenzoylaminolauronic acid, (II), m. 71–2°;  $[\alpha]_{\rm p}^{190}$ —69.1° in EtOH. E. E. GORSLINE.

Triazo Group. XVIII. β-Triazoethylamine. M. O. Forster and S. H. New-MAN. Royal Coll. of Science, London. Proc. Chem. Soc., 27, 154; J. Chem. Soc., 99, 1277-82.—\$\textit{\textit{F}} Triazoethylamine, \text{N}\_2(CH\_2)\_2\text{NH}\_2, is obtained from the product of heating β-bromoethylamine hydrobromide with NaN<sub>2</sub>; colorless oil, b<sub>80</sub> 63.5°. Hydrochloride, needles, m. 72°. Benzoyl deriv., oil. It is reduced to ethylenediamine by Sn and HCl. β-Triazoethylphthalimide, C<sub>2</sub>H<sub>4</sub>: (CO)<sub>2</sub>: N(CH<sub>2</sub>)<sub>2</sub>N<sub>2</sub>, is obtained from the product of heating bromoethylphthalimide with NaNa; pale yellow needles, m. 58°. By heating an alc. soln. of p-nitrobenzaldehyde with the calculated quant. of triazoethylamine hydrochloride neutralized by Na<sub>2</sub>CO<sub>2</sub>, p-nitrobenzylidene-β-triazoethylamine, NO<sub>2</sub>C<sub>3</sub>-H<sub>4</sub>CH : N(CH)<sub>2</sub>N<sub>2</sub>, results; rectangular plates, m. 71°. From p-toluenesulfonyl chloride and β-triazoethylamine, in pyridine, p-toluene-β-triazoethylsulfonamide, MeC<sub>2</sub>H<sub>4</sub>SO<sub>2</sub>NH(CH<sub>2</sub>)<sub>2</sub>N<sub>3</sub>, is obtained, needles, m. 64°. β-Triazoethylcarbamide, N<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>-NHCONH<sub>2</sub>, flakes, m. 59°. s-Phenyl-β-triazoethylcarbamide, N<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>NHCONHPh, needles, m. 99°. s-Phenyl-β-triazoethylthiocarbamide, C<sub>2</sub>H<sub>11</sub>N<sub>4</sub>S, rhomboidal plates, m. 64°. From triazoethyl iodide and quinoline, after 5 ds. in the dark, β-triazoethylquinolinium iodide, C<sub>2</sub>H<sub>7</sub>NI(CH<sub>2</sub>)<sub>2</sub>N<sub>2</sub>, is obtained; golden yellow prisms, becoming deep red at 162°, m. 162-4° (decomp.). Chloroplatinate, C. H., N. Cl. Pt., pale brown needles, m. 200°. E. E. GORSLINE.

Syntheses with Phenol Derivatives Containing a Mobil Nitro Group. IV. Quinone-imides; Asymmetric Quaternary Ammonium Compounds and Asymmetric Carbinols. R. Meldola and H. Kuntzen. Finsbury Tech. Coll. Proc. Chem. Soc., 27, 157; J. Chem. Soc., 99, 1283-1302.—From the iminazoles obtained by the action of primary amines on trinitroacetylaminophenol, iminoazolium iodides, (I), can be obtained by heating with alkyl halides. These compds. can be hydrolyzed giving iminazolium hydroxides. The latter lose H<sub>2</sub>O on heating and quinoneimides, (II), result. On soln. in alkali these are transformed into the isomeric carbinols, (III). As the carbinols

and aminazolium hydroxides are interconvertible, the possibility is offered of transferring optical activity from a N to a C atom. (II) represents a new type of quinone-imide derivs. which are named iminazolones. 4,7-Dinitro-6-hydroxy-1-phenyl-2,3-dimethylbensiminazolium iodide, C<sub>18</sub>H<sub>12</sub>O<sub>8</sub>N<sub>4</sub>I, red cryst. powder, decompd. about 242-4°. 4,7-Dinitro-6-hydroxy-1-phenyl-2,3-dinitrobensiminazolium hydroxide, flat red needles, cryst. with 1 mol. of attached H<sub>2</sub>O. Even in a desiccator the compd. undergoes complete hydration, passing into the quinoneimide in 2 or 3 ds. 4,7-Dinitro-6-hydroxy-1-phenyl-2,3-dimethylbensiminazole, C<sub>18</sub>H<sub>14</sub>O<sub>8</sub>N<sub>4</sub>, is an ocherous, micro-

4,7-Dinitro-6-ethoxy-1-phenyl-2-methylbenziminazole, 'C1eH14O4N4, flat, cryst. compd. needles, m. 179°. 4,7-Dinitro-6-hydroxy-1-phenyl-2,3-dimethylbenziminasolium chloride, C, H, O, N, Cl, results by adding the base to a cold satd. soln. of HCl in EtOH; microscopic needles. Chloroplatinate, (C18H12O8N4)2.H2PtCl4.2H2O; ocherous plates. Ferrichloride, light ocherous needles. Nitrate, white prisms. Acetate, golden scales, decomp. 286-7°. 4,7-Dinitro-6-hydroxy-1-phenyl-2-methyl-3-ethylbenziminasolium hydroxide, C10H16O2N4, bright red, flat needles, decomp. about 250-8°. By heating the above compd. to 110° 4,7-dinitro-6-hydroxy-1-phenyl-2-methyl-3-ethylbenziminazolone results. It does not differ in appearance from the iminazolium hydroxide. 4,7-Dinitro-6-hydroxy-1-phenyl-2-methyl-3-ethylbenziminazolium iodide, red ocherous needles. Chloroplatinate, ocherous, amorphous. The corresponding benziminazolol, CapHaOoNA is a nonbasic microcryst., ocherous powder. 4,7-Dinitro-6-hydroxy-1-p-tolyl-2,3dimethylbenziminazolium hydroxide, C10H1002N4, ocherous scales. Iodide, dark and light ocherous cryst. nodules. The corresponding benziminazolone, C1. H1. O.N., is a microcryst., ocherous substance. Picrate, pale vellow needles, m. 176°. The bensiminazolol, C18H18O2N8, is an ocherous microcryst. powder, decomp. above 300°. E. E. GORSLINE.

Triketohydrindene Hydrate. IV. Hydrindantin and its Analogs. S. RUHEMANN. Cambridge. Proc. Chem. Soc., 27, 163; J. Chem. Soc., 99, 1306-10.—Hydrindantin yields a red soln. with Na<sub>2</sub>CO<sub>2</sub> and a blue soln. with NaOH (C. A., 5, 3051). The red salt is a salt of hydrindantin and the blue Na compd. is a chromo salt of a hydrolytic product of hydrindantin, hydroxydiketohydrindene, CaH4: (CO)s: CHOH. compd. is the analog of dialuric acid. It could not be obtained pure, readily oxidizing to hydrindantin. The violet Ba compd. yielded by hydrindantin is a chromo salt of dialuric acid. Isatide seems to be an analog of alloxantin and should be represented thus:

(Heller, Ber., 37, 946 (1904)). Dialuric acid does not unite with triketohydrindene hydrate but reduces the later to hydrindantin. This compd. also results from the treatment of oximinodiketohydrindene with conc. HI. On using SnCl, instead of the acid the analog of uranil, diketohydrindamine, CoH4: (CO)2: CHNH2, is formed. E. E. GORSLINE.

Synthesis of 4,6-Dimethoxy-2-β-methylaminoethylbenzaldehyde. A. H. Salway. Wellcome Chem. Research Lab. Proc. Chem. Soc., 27, 191; J. Chem. Soc., 99, 1320-5.  $-\beta$ -3,5-Dimethoxyphenylpropionamide, (I), is prepared from the corresponding acid

(C. A., 5, 1597) by the successive action of PCl<sub>4</sub> and NH<sub>2</sub>; colorless needles from C<sub>4</sub>H<sub>6</sub> + light petroleum, m. 80-1°. When fused, after solidification, m. 86°. When this compd. is treated with NaOCl and an Et,O extract of the product with phenylacetyl chloride, there results phenylacetyl-β-3,5-dimethoxyphenylethylamide, C<sub>18</sub>H<sub>m</sub>O<sub>2</sub>N, prismatic needles from dil. EtOH, m. 73°. When treated with P4O10 this compd. yields 6.8-Dimethoxy-I-benzyl-3,4-dihydroisoquinoline, (II), viscid oil. Hydrochloride, rhombohedral prisms with 2 H<sub>2</sub>O, m. 181° (decomp.). Picrate, yellow prisms, m. 184°. By conversion into its methochloride and subsequent reduction by Sn and HCl, the above compd. vields 6,8-dimethoxy-I-benzoyl-2-methyl-I,2,3,4-tetrahydroisoquino-

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line, (III), m. 52°. Picrate, feathery needles from EtOH, decomp. 195°. Upon oxidizing this compd. in dil. H<sub>2</sub>SO<sub>4</sub> with pytolusite, 4,6-dimethoxy-2-β-methylamino-

ethylbensaldehyde, (IV), results; gummy solid. Hydrochloride, needles with 2H<sub>4</sub>O, m. when anhyd. 164°. Picrate, yellow needles, m. 155-6°. Chlorocurate, golden leaf-lets from EtOH, m. 131-2°

E. E. GORSLINE.

Indicators of the Methyl-red Type. H. HOWARD AND F. G. POPE. Royal Coll. of Science, London. J. Chem. Soc., 99, 1333-6.—The sodium salt of methyl-red (Tizard, C. A., 5, 1222) was prepared by grinding the compd. with an insufficient quant, of 10% NaOH to convert it into the salt. After drying in a vacuum over H.SO. the mass was extracted with b. toluene and again dried; scarlet plates. Potassium salt, similarly made, dark red plates. By combining diazotized anthranilic acid with the required amt. of diphenylamine dissolved in 30 times its wt. of alc., keeping on ice for 2 hrs. and then heating to 40° for 2 hrs., o-carboxybenzeneasodiphenylamine. HO<sub>2</sub>CC<sub>2</sub>H<sub>4</sub>N<sub>2</sub>C<sub>2</sub>H<sub>4</sub>NHPh, resulted; olive-green needles, m. 231°. Sodium salt, prepared as described above, dark red needles giving an orange-colored soln. in H4O; m. 231°. o-Carbethoxybenzeneaso-α-naphthylamine, HO<sub>2</sub>CC<sub>2</sub>H<sub>4</sub>N<sub>2</sub>C<sub>10</sub>H<sub>4</sub>NH<sub>20</sub> similarly prepared, green prisms, m. 243°. Sodium salt, red needles, giving a red soln, in H.O. o-Carboxybenzeneazophenyl-a-naphthylamine, HO,CC,H,N,C10H,NMe2, dark green needles m. 196°. Sodium salt, dark reddish brown solid, somewhat deliquescent. Using satd. alc. solns. of these compds. as indicators, o.o. N solns. of NaOH and Na<sub>2</sub>CO<sub>2</sub> were titrated with acids 2 drops of indicator being used to 10 cc. of alk, soln. The end points were more sharply defined in each case than when methyl orange is employed. E. E. GORSLINE.

Dihydrocinnamenylcarbimide (β-Phenylethyl Isocyanate). M. O. FORSTER AND H. STÖTTER. Royal Coll. of Science, London. J. Chem. Soc., 99, 1337-40.—By heating dihydrocinnamyl chloride with NaN<sub>2</sub>, in dry toluene, for 2 d., dihydrocinnamenylcarbimide (β-phenylethyl isocyanate), PhCH<sub>2</sub>CH<sub>2</sub>N: CO, was formed; mobil liquid, b<sub>13</sub> 109-11°. By treating this compd. with PhNH<sub>2</sub>, dihydrocinnamenylphenylcarbamide, PhCH<sub>2</sub>CH<sub>2</sub>NHCONHPh, results; lustrous leaflets from C<sub>2</sub>H<sub>4</sub>, m. about 148°. From the isocyanate and PhN<sub>2</sub>H<sub>2</sub>, s-dihydrocinnamylphenylsemicarbaside, C<sub>10</sub>H<sub>17</sub>ON<sub>4</sub>, results; lustrous needles from EtOH, m. 216°. Menthyl dihydrocinnamenylcarbamate, PhCH<sub>2</sub>CH<sub>2</sub>NHCO<sub>2</sub>C<sub>10</sub>H<sub>10</sub>, silky needles from dil. acetone, m. 86°; [α]<sub>0</sub> —65° in absol. EtOH.

Constitution of Berberine. C. K. Tinkler. Univ. Birmingham. Proc. Chem. Soc., 27, 162; J. Chem. Soc., 99, 1340-7.—The ultraviolet absorption spectra of berberine and its simple derivs. have been examined. There is evidence of the existence of 2 modifications of the alkaloid in the solid state. Berberine cryst. from H<sub>2</sub>O gives spectra in aq. alc., and CHCl<sub>2</sub> solns. identical with these of the berberine salts. From this the substance would appear to have the ammonium constitution (I) (Perkin,

$$(MeO)_{\mathfrak{g}}C_{\mathfrak{g}}H_{\mathfrak{g}} \underbrace{CH: C}_{CH: N(OH)CH_{\mathfrak{g}}CH_{\mathfrak{g}}}C_{\mathfrak{g}}H_{\mathfrak{g}}: O_{\mathfrak{g}}: CH_{\mathfrak{g}}$$

C. A., 4, 1613). A soln. containing alkali, however, gives spectra which agree closely with those of Freund and Beck's methyldihydroberberine (Bsr., 37, 4677 (1904)),

and the same spectra are given by a CHCl<sub>2</sub> and an Et<sub>2</sub>O soln. of berberinal, the compd. pptd. by adding NaOH to an aq. soln. of berberinium hydroxide. This can only be explained by assigning to berberinal the formula (II) and for this compd. the name

$$(MeO)_{2}C_{6}H_{2} \leftarrow CH_{CH(OH)} \cdot N \cdot CH_{2} \cdot CH_{2} \rightarrow C_{6}H_{2} : O_{3} : CH_{2}$$

$$(II)$$

berberinol is suggested. Evidence is obtained that dihydroberberine and oxyberberine are formed when berberinol is decompd. by an excess of alkali. Since both modifications of the alkaloid can be obtained in soln. in CHCl<sub>2</sub>, it appears that both the ammonium base and the carbinol exist in the solid state. In this respect berberine differs from the other compds. investigated in connection with the carbinol-ammonium base—aldehydeamine isomerism, where evidence of the existence of the carbinol form alone in the solid state has been obtained.

E. E. G.

Cupriglycolates. S. U. PICKERING. Harpenden. Proc. Chem. Soc., 27, 192; J. Chem. Soc., 99, 1347-53.—The detn. of the mol. wts. of 6 compds. showed that the cupri-compds. formed by the action of alkalies on Cu salts have their Cu in the form of CuO or CuOH, and not as metal displacing H ats. (C. A., 5, 2068). More conclusive evidence has been obtained by finding that in K cupriglycolate the ratio of Cu: K is 1:1, in accordance with the formula CH<sub>2</sub>O(CuOH)CO<sub>2</sub>K, and not 1:2 as it would have been if the metal displacement H: (CH<sub>2</sub>O)<sub>2</sub>Cu(CO<sub>2</sub>K)<sub>2</sub>. From a soln. of Cu glycolate mixed with excess of KOH, alc. ppts. a mauve-colored, alkaline, cryst. compound, HOCH<sub>2</sub>C(OK)(OH)Cu<sup>IV</sup>(OH)(OK)CO<sub>2</sub>CH<sub>2</sub>OH, agreeing in properties and comp. with the \(\gamma\)-cupri-salts (containing the cuprite group) suggested as existing in such solns. The corresponding salts of Na, Rb and probably Ba have been obtained, but not those of Li, Ca or Cs. On heating, they seem, in some cases, to be transformed into compds. in which the at. of Cu displaces 2 ats. of H in alc. OH groups.

E. E. G.

Synthesis of Derivatives of Thioxanthone from Aromatic Disulfides. E. G. MARSDEN AND S. SMILES. Univ. Coll., London. J. Chem. Soc., 99, 1353-8.—The methods of synthesizing thioxanthones from disulfides may be said to fall under either of 2 types: (1) The CO<sub>2</sub>H, which is to form the CO of the thioxanthrone is present in the disulfide chosen, and in this case, the starting material may be di-o-thiobenzoic acid or its derivs., which are submitted to condensation with any aromatic compd. (2) The CO<sub>2</sub>H is present in the aromatic compd. Here any suitable disulfide may be condensed with suitably substituted derivs. of BzOH; for example, m-hydroxybenzoic acid. By dissolving di-o-thiobenzoic acid in conc. H<sub>2</sub>SO<sub>4</sub>, adding an excess of p-xylene and heating to 50% for 45 min. there results, 1,4-dimethylthioxanthone, (I), yellow needles

$$C_{\bullet}Me$$

$$C_{\bullet}H_{\bullet} C_{\bullet}C_{\bullet}H_{\bullet}Br$$

$$C_{\bullet}H_{\bullet} CO$$

$$C_{\bullet}H_{\bullet}COEt$$

$$(II)$$

$$(III)$$

$$(IIII)$$

from EtOH, m. 112°. When m-xylene is employed, 1,3-dimethylthioxanthone, C<sub>18</sub>H<sub>12</sub>OS, results; silky, yellow needles, from EtOH, m. 127°. PhBr and di-o-thiobenzoic acid give bromothioxanthone, (II), yellow needles from EtOH, m. 141°. Using p-chlorophenetole, chlorothioxythioxanthone, (III), results; yellow needles from EtOH, m. 144°. By using p-chlorophenol and the same acid, chlorohydroxythioxanthone, C<sub>18</sub>H<sub>1</sub>O<sub>3</sub>ClS, results; yellow needles, m. 253°. Sodium salt, orange needles. A soln. of equimol. proportions of m-hydroxybenzoic acid and di-p-thiodimethylaniline, in

conc. H<sub>2</sub>SO<sub>4</sub>, is heated to 60-70°. The product is poured into ice, treated with Na<sub>2</sub>CO<sub>8</sub> and the solid product, formed on standing, treated with hot alc. The insol. portion is recrystd. from b. phenetole; the dimethylaminophenylthiol deriv. of hydroxydimethylaminothioxanthone, C<sub>28</sub>H<sub>28</sub>O<sub>2</sub>N<sub>2</sub>S<sub>3</sub>, results; garnet prisms, m. 250°. Chloroplatinate, insol. yellow powder of high m. p. Sodium salt, scarlet, insol. in cold H<sub>2</sub>O. On adding H<sub>2</sub>O and a little AcOH to the portion sol. in alc. an isomeride of the above thioxanthone containing I H<sub>2</sub>O results, C<sub>28</sub>H<sub>28</sub>O<sub>2</sub>N<sub>2</sub>S<sub>3</sub>.H<sub>2</sub>O; orange powder. The sodium salt and hydrochloride are sol. in H<sub>2</sub>O. The H<sub>2</sub>O is not completely expelled at 150° when decomp. commences.

Relation of the Velocity of Chlorination of Aromatic Compounds to Constitution.

I. Chlorination of Anilides. K. J. P. Orton and H. King. Univ. Coll. of N. Wales.

Proc. Chem. Soc., 27, 196; J. Chem. Soc., 99, 1369-77.—The velocity of chlorination—
formation of monochloro derivs.— of a number of acylanilides has been measured in
glac. AcOH soln. The rate of reaction varies enormously with the character of the
substituent in the C<sub>2</sub>H<sub>6</sub> nucleus. The nature of the acyl groups has a comparatively
subsidiary effect. Naphthalides are chlorinated at a far greater rate than the anilides.

E. E. G.

Chlorination of Acylanilides. Effect of the Constitution of the Acyl Group on the Proportion of the o- and p-Derivatives. H. KING AND K. J. P. ORTON. Univ. Coll. of N. Wales. Proc. Chem. Soc., 27, 196; J. Chem. Soc., 99, 1377-82.—"The proportion of o- and p-chloro derivs. produced in the chlorination of formic, propionic, stearic and benzoic anilides has been estimated. Comparison with acetanilide (Jones and Orton, C. A., 3, 2805) shows that the largest proportion of the o-chloro deriv. is formed in this case. Acetanilide yields 45, propionic anilide 26, stearic anilide 12, benzoic anilide 11, and formic anilide 3% of the o-chloro deriv."

E. E. G.

Purification and Properties of Acetic Acid. W. H. BOUSFIELD AND T. M. LOWRY. Proc. Chem. Soc., 27, 187; J. Chem. Soc., 99, 1432-41.—AcOH may be purified by distilling from KMnO4, using a still head to retain acids of higher b. p., and then freezing to remove the  $H_2O$ . The purified acid m.  $16.6^\circ$ ;  $d_4^{10}$  1.05148;  $d_4^{20}$  1.04922. Its max. cond. when mixed with  $H_2O$  is  $K_{10}$  0.001641<sub>3</sub>. E. E. G.

Some Oxidation Products of the Hydroxybenzoic Acids. III. A. G. PERKIN. Leeds Univ. Proc. Chem. Soc., 27, 194; J. Chem. Soc., 99, 1442-50.—The comp. C<sub>14</sub>H<sub>4</sub>O<sub>11</sub>, called ceruleoellagic acid, obtained by heating ellagic or flavellagic acid with H<sub>2</sub>SO<sub>4</sub> (Proc. Chem. Soc., 22, 114 (1906)) can also be prepared by the action of H<sub>2</sub>AsO<sub>4</sub> on the H<sub>2</sub>SO<sub>4</sub> solns. of these compds. Acetyl derivative, C<sub>14</sub>H<sub>10</sub>(Ac)<sub>6</sub>, colorless, m. 330-2°. Benzoyl derivative, needles, m. 343-5°. When distilled with Zn dust, fluorene results, and with b. KOH soln. a compd., C<sub>12</sub>H<sub>10</sub>O<sub>6</sub>, is formed, crystg. in prismatic needles and evidently an octahydroxydiphenyl. Acetyl derivative, C<sub>12</sub>H<sub>2</sub>O<sub>6</sub>(Ac)<sub>6</sub>; colorless needles, m. 177-8°. Ceruleoellagic acid dyes mordanted fabrics similarly to flavellagic acid, but somewhat more strongly.

E. E. G.

Interaction of Formic Acid and Cellulose. C. F. Cross and E. J. Bevan. Proc. Chem. Soc., 27, 149; J. Chem. Soc., 99, 1450-6.—Derivs. of cellulose obtained by direct interaction with HCO<sub>2</sub>H and also in the presence of catalysts have been examined. The quant. data as to composition and decomp. are not reconcilable with the view that the products are simple esters of a normal cellulose. Besides the normal replacement of alc. OH by the OCOH residue, the acid, owing to its avidity, could attack the cellulose complex by hydrolysis or condensation, or by both processes concurrently. It might also react through the CO, O at. These modes of reaction may explain the observed anomalies. The interaction of resorcinol and HCO<sub>2</sub>H in the presence of HCl yields a cryst. yellow product, C<sub>12</sub>H<sub>12</sub>O<sub>6</sub>, suggesting a dihydroxybenshydrol. Alk.

solns. of this compd. are crimson in color. Pentaacetyl derivative, crysts. from EtOH. Quinol gives no product suggesting that the condensation takes place in the p-position with respect to an OH.  $\alpha$ -Naphthol reacts with the acid while  $\beta$ -naphthol does not. Phloroglucinol reacts rapidly, but the first product appears to be a phloroglucide. E. E. G.

Structure of Xanthonium and Acridinium Salts. J. T. Hewitt and F. B. Thole. Proc. Chem. Soc., 26, 265.—Phenylxanthenol and phenylmethylacridol give nearly the same absorption spectra. Acids effect a change, pointing to the salts formed having similar constitutions. Since the view that MeI forms an additive product with phenyl-

acridine of the constitution CPh  $C_eH_eI$  NMe seems untenable (Kehrmann, C. A.,

4, 2130), the authors prefer the oxonium structure for the salts obtained from xanthenol and its derivs.

E. E. G.

Iodoacenaphthene. H. CROMPTON AND M. K. HARRISON. Proc. Chem. Soc., 26, 226.—Acenaphthene is dissolved in hot EtOH, cooled slightly, 1 mol. I added and somewhat more than the calculated quant. of yellow HgO gradually added. The mixture is raised to the b. p. and filtered. There results iodoacenaphthene, C<sub>12</sub>H<sub>0</sub>I, needles or prisms, m. 65.5°, decompg. about 180°. It is best purified as the picrate, orange-red needles, m. 102.5°. On oxidation with Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in AcOH soln. an iodonaphthalic acid results, the anhydride of which m. 231°.

E. E. G.

Synthesis of Nitrognoscopine and Allied Substances. E. HOPE AND R. ROBINSON. Proc. Chem. Soc., 26, 228.—By b. equimol. quants. of cotarnine and nitromeconine in alc. soln. for a few min., nitrognoscopine, CH<sub>2</sub>: O<sub>2</sub>: C<sub>18</sub>H<sub>11</sub>O<sub>2</sub>(OMe<sub>3</sub>)NO<sub>3</sub>, is obtained, bright yellow prisms crystg. from trichloroethylene, m. to a red liquid 191° (decomp.). Hydrochloride, chloroaurate, picrate and methiodide, yellow, m. 205-6°. By reduction with Sn and HCl, aminognoscopine is formed; prisms, m. 205°; decompd. by b. glacial AcOH. Acetylaminognoscopine, needles, m. 198°. Nitro-ψ-gnoscopine, from cotarnine and nitro-ψ-meconine, yellow needles, m. 184°. Anhydrocotarninenitrophthalide (nitrodesdimethoxygnoscopine), CH<sub>2</sub>: O<sub>2</sub>: C<sub>18</sub>H<sub>11</sub>O<sub>2</sub>(OMe)NO<sub>2</sub>, prisms, m. 166-7°; readily decompd. by hot glacial AcOH. Anhydrocotarnineaminophthalide, rectangular plates, m. 216-8°.

Complete Methylation by Methyl Sulfate. R. MELDOLA. Proc. Chem. Soc., 26, 232.—Isopicramic acid was treated with alkali and Me<sub>2</sub>SO<sub>4</sub>, using a quant. of the latter a little in excess of the amt. required to introduce 1 Me. The unmethylated portion was largely the unchanged acid while the methylated portion was 2,6-dinitrodimethyl-p-anisidine, C<sub>6</sub>H<sub>2</sub>(NO<sub>2</sub>)<sub>2</sub>OMeN(Me<sub>2</sub>), orange needles, m. above 300°. E. E. G.

Interaction of Phenylmercaptan and Thionyl Chloride. H. S. TASKER AND H. O. JONES. Proc. Chem. Soc., 26, 234.—The conclusions reached by the authors concerning the reaction (C. A., 4, 1023) have been criticized by Smythe and Foster (C. A., 4, 2938) who state that they see no reason why the reaction is not completely represented by the equation of Holmberg (C. A., 2, 1690). The authors claim that S. and F. overlook the following facts: (1) The liquid product of the reaction has the properties and composition of diphenyl tetrasulfide; (2) when SOCl<sub>2</sub> acts with Pb phenylmercaptide or with Ni carbonyl, SO<sub>2</sub> is formed and in the latter case S also. In both cases SO<sub>2</sub> appears to be formed by the decomp. of S suboxide; (3) H<sub>2</sub>O is always present at the end of the reaction and therefore all of the SO<sub>2</sub> can not be due to the secondary interaction between H<sub>2</sub>O and SOCl<sub>2</sub>.

Formation of a Six-membered Ring through the Agency of the Imino Group. F. B.

THOLE AND J. F. THORPE. Proc. Chem. Soc., 26, 295.—An open chain of 5 C ats. and 1 N at. passes into a 6-membered ring through the agency of the imino group. When acetone is condensed with cyanoacetamide in the presence of pyradine or NaOH, an almost quant. yield of the iminoimide (I) results, and when MeEt ketone is used an equally good yield of the iminoimide (II) is obtained. These compds. are tautomeric

iminoamino (ketimino-enamic) derivs., which are quickly hydrolyzed by dil. mineral acids forming the corresponding imides. Either from the iminoimides or from the imides a quant. yield of the  $\beta,\beta$ -substituted glutaric acid is formed on acid hydrolysis. The iminoimides undergo a further imino condensation when treated with NaOEt, yielding the diiminodiimides (III) and (IV). These compds. are tautomeric diimino-

diamino compds. which are readily hydrolyzed by dil. mineral acids, forming the corresponding diimides. The latter are converted into the corresponding dialkylglutaric acids on complete acid hydrolysis.

E. E. G.

New Syntheses of Thioxanthone and its Derivatives. S. SMILES. Proc. Chem. Soc., 26, 342.—The following methods of synthesis are available for the formation of these compds.: (1) Condensation of dithiobenzoic acid with aromatic derivs in  $H_2SO_4$ ; (2) condensation of m-hydroxy- or m-aminobenzoic acids with (a) suitable disulfides, or (b) certain sulfinic acids in  $H_2SO_4$ ; (3) action of o-thiolbenzoic acid on certain quinones and elimination of  $H_2O$  from the product; (4) condensation of aromatic compds. with either o-carboxylbenzenesulfinic acid or with o-thiobenzoic acid (Davis and Smiles, C. A., 4, 3073).

Syntheses of 3- $\beta$ -Aminoethylindole and its Formation from Tryptophan. A. J. Ewins and P. P. Laidlaw. *Proc. Chem. Soc.*, 26, 343.—This compd. has been made by heating together  $\gamma$ -aminobutyrylacetal and phenylhydrazine in the presence of ZnCl<sub>2</sub>. It has also been obtained by submitting tryptophan to the action of putrefactive organisms. E. E. G.

Empirical Relation between the Configuration and Rotation of Sugars. Ernest Anderson. Kent Chem. Lab., Univ. Chicago. J. Am. Chem. Soc., 33, 1510-4.— In all cases where both configuration and rotation are known, it is found that the configuration of the  $\alpha$ - and  $\beta$ -C ats. determins the direction and magnitude of the rotation.

levo, —C C CX and —C C CX slightly (less than 20°) l or d. Hence the H H O OH OH O

configuration of many sugars may be determined if their rotation be known.

S. J. BATES.

Reaction between Organic Magnesium Compounds and Cinnamylidene Esters. Grace Potter Reynolds. Barnard Coll. Am. Chem. J., 46, 198-211; C. A., 1, 2887; 3, 169; 5, 83.—The methods of preparing stable and allocinnamylideneacetic

acids were improved. The methyl ester of the allo acid solidifies about  $-15^{\circ}$  to a white crystal and is partly changed on distillation into the methyl ester of the stable acid; both esters decomp. on standing. The stable ester and PhMgBr (2.5 mols.) yield  $\beta$ -phenyl- $\gamma$ -benzalpropyl phenyl ketone, which with 2 mols. PhMgBr at room temp. gives a quant. yield of  $I_1,I_3,5$ -tetraphenyl-4-pentene-I-ol, PhCH: CHCHPhCH<sub>2</sub>CPh<sub>3</sub>OH, fine white crystals from alc., m. 134. On oxidation with KMnO<sub>4</sub> in acetone, this alc.

yields BzOH and \(\gamma\text{-kydroxytriphenylbutyric lactone}\), Ph.CCH,CHPhCOO, long, iridescent needles from alc., m. 157°. Using 4 mols. PhMgBr, the allo ester, and less readily the stable ester yield  $\beta$ -phenyl- $\gamma$ -benzalpropyl phenyl ketone. The ester of the unstable acid with PhCH, MgBr yields (a) \(\beta\)-benzyl-\(\gamma\)-benzyl benzyl ketone, PhCH: CHCH-(CH<sub>2</sub>Ph)CH<sub>2</sub>COCH<sub>2</sub>Ph, lemon-yellow liquid, b<sub>18</sub> 265°. Dibromo-β-benzal γ-benzalpropyl ketone, white crystals from ligroin + CHCl<sub>2</sub>, m.  $165-5^{\circ}$ . (b)  $\alpha, \alpha$ -dibensylδ-benzalcrotonyl alcohol, PhCH: CHCH: CHC(CH,Ph),OH, mobil lemon-yellow liquid, b<sub>10</sub> 200°. Tetrabromo-a, a-dibensyl-d-bensalcrotonyl alcohol, fine needles from CHCl<sub>2</sub>, m. 227°. Alc. KOH on the original mixture of (a) and (b) gives β-bensyl-γbanzalbutyric acid, PhCH: CHCH(CH,Ph)CH,CO,H, heavy iridescent plates from acetone + ligroin, m. 144°. Methyl ester, crystals from MeOH, m. 66°. The acid on oxidation with KMnO, yields BzOH and benzylsuccinic acid. With EtMgBr the ester of the stable acid yields (a) a, a-diethyl-3-benzalcrotonyl alcohol, pale yellow liquid, b<sub>10</sub> 169° and the secondary products, (b) ester, fine needles, m. 136°, which with KOH gives an acid, m. 230°. (c) KOH on the original product gave an acid, fine needles, m. 179°. The yield of (a) is best at low temp.

Action of Hydrazoic Acid on Carbylamines. E. OLIVERI-MANDALÀ AND B. ALAGNA. Gazz. chim. ital., 40, II, 441-4; Chem. Zenir., 1911, I, 662; cf. C. A., 4, 2455.—Like methylcarbylamine, the Et and Ph compds. can be converted into the corresponding tetrazole derivs. N-Methyltetrazole is a very weak base; hydrochloride, prismatic needles, takes up H<sub>2</sub>O from the air and decomp. N-Ethyltetrazole, b<sub>14</sub> 155-6°. Chloroplatinate, yellow powder, decomp. above 160° without m. Chas. A. Rouiller.

Influence of Alkyl Substituents on the Electrical Conductivity of Malonic Acids. WILLIAM BUELL MELDRUM. McGill Univ., Montreal. J. Phys. Chem., 15, 474-88. -The elec. cond. of alkyl derivs. of malonic acid was measured at 25° up to V 1024 and the dissociation const. detd. from the cond. of the acid Na salt at V 1024. Below are the values obtained for  $K \times 10^{8}$  (only the substituent alkyl groups are given): Malonic, 0.158; Me, 0.087; Et, 0.127; Pr, 0.115; Bu, 0.116; Me, CH, 0.135; Me, CHCH, 0.101; Me<sub>2</sub>CHCH<sub>2</sub>CH<sub>2</sub>, 0.131; MePr, 0.212; EtPr, 1.160; Pr<sub>2</sub>, 1.186; (Me<sub>2</sub>CH)Me, 0.141; (Me,CH)Et, 1.284; (Me,CHCH,)Me, 0.353; (Me,CHCH,)Et, 1.860; (Me,CHCH,CH,)Me, o.210; (Me<sub>2</sub>CHCH<sub>2</sub>CH<sub>2</sub>)Et, 1.020; Me<sub>2</sub>, 0.080; MeEt, 0.161; MeBu, 0.203; EtBu, 1.163; Et. 0.751. A single substituent decreases the dissociation const.; as the number of C ats. in normal substituents increases the const. approaches that of the nonsubstituted acid, but the iso derivs. do not show the same regularity. In general, acids containing isoalkyl groups are dissociated to a greater extent than their normal isomers; the same is true of the dialkyl as compared with monoalkyl acids, the second alkyl group raising the const, above that of the nonsubstituted acid itself. Since, in general, in dibasic acids, the dissociation of 1 CO<sub>2</sub>H group is affected by the proximity of the other, it would seem that the introduction of 1 alkyl group interferes in the interaction between the 2 CO<sub>4</sub>H groups, while a second alkyl causes a further change in configuration of the mol.; either the 2 CO<sub>2</sub>H groups are actually brought closer together or the space between them is left free of interfering groups.

Synthesis of Pentamethylenediguanidine. OTTO RIPEE. Physiol. Inst., Univ. Heidelberg. Z. physiol. Chem., 72, 484-5.—Cyanamine was allowed to act on penta-

methylendiamine for 17 days and then treated according to Kossel's method for the prep. of agmatine. Pentamethylenediguanidine sulfate was obtained, difficultly sol. in H<sub>2</sub>O, not m. 300°. Chloroaurate, m. 161°. G. M. MEYER.

Nitroclupeine. A. Kossel and E. L. Kennaway. Physiol. Inst., Univ. Heidelberg. Z. physiol. Chem., 72, 486-91.—Clupeine can be converted into a nitro compd. which on hydrolysis yields nitroarginine, m. 227-8°, d-rotatory and identical with the substance obtained by nitration of arginine. The NO<sub>3</sub> has very probably attached itself to the guanidine radical, from which it would follow that the guanidine radical is unattached and not involved in the peptide linkage. Nitroclupeine, white powder, obtained by the nitration of clupeine sulfate, is purified by dissolving in 2% NaOH and repptg. with H<sub>2</sub>SO<sub>4</sub>. This compd. gives the biuret reaction similar to other protamines.

G. M. M.

Arginine Salts. F. Weiss. Physiol. Inst., Univ. Heidelberg. Z. physiol. Chem., 72, 490-3.—d,l-Arginine chloroaurate, C<sub>6</sub>H<sub>14</sub>O<sub>4</sub>N<sub>2</sub>.2HCl, AuCl<sub>2</sub>.0.5H<sub>2</sub>O, m. 105-15°. d-Arginine chloroaurate, C<sub>6</sub>H<sub>14</sub>O<sub>2</sub>N<sub>4</sub>.2HCl.2AuCl<sub>2</sub> + 1.5H<sub>2</sub>O, softens 140°; m. above 160°. Loses its H<sub>2</sub>O in vacuo at ordinary temp., but more readily at 40-5°. Arginine sulfate, C<sub>6</sub>H<sub>14</sub>O<sub>2</sub>N<sub>4</sub>.H<sub>2</sub>SO<sub>4</sub>.H<sub>2</sub>O. dl-Arginine forms 2 kinds of salts with H<sub>2</sub>SO<sub>4</sub>. Only one could be suitably isolated for analysis. This decomposes when heated to 100°. G. M. M.

Action of Hypochlorous Acid on Ethylenehydrocarbons. A. Umnova. Univ. for Women, St. Petersburg. J. Russ. Phys. Chem. Soc., 42, 1530-43.—Dimethylpropylene when cooled and treated with the calculated amt. of HClO forms the chloride Me<sub>2</sub>CHCHClCMe: CH<sub>2</sub>, b<sub>20</sub> 44-45°, d<sup>20</sup> 0.9083. Similarly diisobutylene forms the chloride Me<sub>2</sub>C.CHCl.Me: CH<sub>2</sub>, b<sub>20</sub> 53-54°, d<sup>20</sup> 0.9042, n<sup>20</sup> 1.4473. Both chlorides are colorless liquids with a pleasant odor; they add Br readily. When treated with a 10% KOH soln. they give the unsatd. alcohols C<sub>7</sub>H<sub>14</sub>O (after 120 hrs.), b. 154-156°, d<sup>20</sup> 0.8427 and C<sub>8</sub>H<sub>15</sub>O (after 600 hrs.), b. 173-175°, d<sup>20</sup> 0.8556, thick liquids, which add Br very readily. Methylisopropylethylene, Me<sub>2</sub>CH.CH: CH.Me, b. 58-59°, d<sup>20</sup> 0.6695 (from the corresponding iodide and alc. KOH) failed to give a pure product on treatment with HClO; the fraction b. 65-70° contained mostly the chlorohydrin, C<sub>9</sub>H<sub>12</sub>ClO. Heating of the impure product with KOH gave pure methylisopropylethylene oxide, C<sub>8</sub>H<sub>12</sub>O, b. 99-100°, which formed the glycol, C<sub>9</sub>H<sub>14</sub>O<sub>9</sub>, on b. with H<sub>2</sub>O; thick liquid, b. 194-196°. In all cases HClO acted the same as Cl. G. Weintraue.

Formation of 1-Nitroso-5-phenyl-3-pyrazolidone from Cinnamoyl Hydrazide. ERNST MUCKERMANN. Chem. Inst., Univ. Heidelberg. J. prakt. Chem., 83, 513-40. -Hydrazides of monobasic unsatd. acids have been hitherto unexamined. The present work deals with cinnamoylhydrazide, PhCH: CHCONHNH, m. 101°, which is readily obtained by b. Et. cinnamate with alc. and N<sub>2</sub>H<sub>4</sub>, H<sub>2</sub>O. It exhibits the usual reducing properties of primary acid hydrazides, yields dicinnamoylkydrazide, N<sub>2</sub>H<sub>2</sub>(COCH: CHPh)<sub>2</sub>, m. 247-8°, by b. with alc. I, and forms a hydrochloride, m. 201° (decomp.), benzylidene compound, PhCH: CHCONHN: CHPh, m. 180°, benzoyl derivative, m. 158-9°, and condensation products with Me<sub>2</sub>CO, PhCH: CHCONHN: CMe, m. 127°, and with Et acetoacetate, PhCH: CHCONHN: CMeCH, CO, Et, m. 125-6°. Its hydrochloride reacts with aq. CNOK to form cinnamoylsemicarbaside, PhCH: CHCONHNHCONH, m. 161-2°. Cinnamoylphenylthiosemicarbaside, PhCH: CHCONHNHCSNHPh, from cinnamoylhydrazide and alc. phenylthiocarbamide, m. 146°. Aqueous cinnamoylhydrazide hydrochloride reacts with NaNO, at o° to form a yellow substance, C.H.O.N., m. 127-8° (decomp.), which is not a hydrazide but a nitroso compd., as is shown by its strongly acid nature and absence of reducing properties. Its acid properties indicate the presence of the group NHCO. The compd. is probably 1-nitroso-5-phenyl-3-pyrazolidone, | NNO, (vide also Knorr, Ber., CO—NH

20, 1107; von Rothenburg, J. prakt. Chem., 51, 140). Anmonium salt,  $C_0H_0O_2N_1NH_3$ , m. 147° (decomp.), obtained from the nitrosophenylpyrazolidone and 10% ammonia, is easily converted into the silver salt,  $C_0H_0O_2N_2Ag$ , m. 144-5°, barium salt,  $(C_0H_1O_2N_3)_2Ba.H_2O$ , greenish blue copper salt,  $(C_0H_0O_2N_3)_2Cu.2H_2O$ , and picrate,  $C_0H_0O_2N_3NH_2.C_0H_2(NO_2)_2OH$ , m. 127°; from the Ag salt ethereal EtI produces *1-nitroso-5-phenyl-2-ethyl-3-pyrazolidone*, m. 98°. The nitrosophenylpyrazolidone yields with Br and glacial AcOH at 0° von Rothenburg's 4,4-dibromo-3-phenyl-5-pyrazolone (J. prakt. Chem., 52, 23). 1-Nitroso-5-phenyl-3-pyrazolidone is converted by b. HCl into cinnamic acid, by cold HCl into  $\beta$ -chlorodihydrocinnamic acid(?), and by b. dil.  $H_2SO_4$ , (d. 1.06), into von Rothenburg's 4-oximino-3-phenyl-5-pyrazolone (loc. cit.).

B. F. P. Brenton.

Ethyl Acetate. A. Kurtenacker and H. Habermann. Tech. Hochsch., Brunn. J. prakt. Chem., 83, 541-52.—Continuing the expts. of Habermann and Brezina (C. A., 4, 905), the authors have investigated the formation of AcOEt from 96 vol. + alc. and glacial AcOH in the presence of NaPO, or the ignited sulfate of Cu, Mg, Ni, Zn, or Na as the dehydrating agent. A mixt. of the alc. and the acid in approx. mol. proportions is kept for some hrs. with a quantity of the dehydrating agent in excess of that required to combine with the H<sub>2</sub>O produced in the reaction; the whole is then heated for several hrs. under a reflux condenser, the course of the reaction being followed by siphoning off samples at intervals, cooling, filtering, and titrating the residual AcOH with 0.5 N NaOH. Sodium metaphosphate or Na<sub>2</sub>SO<sub>4</sub> has no influence whatever on the yield of AcOEt; CuSO<sub>4</sub>, ZnSO<sub>4</sub>, Mg<sub>2</sub>SO<sub>4</sub> produce approximately the same effect, NiSO4 is by far the best dehydrating agent. Having shown that an excess of alc. or prolonged heating of the mixt. does not improve the yield of purified AcOEt, the authors state the following as the best conditions for the prep. of the ester. A mixt. of 200 g. of AcOH and about 160 g. of anhydrous NiSO4 is distilled until the temp. begins to exceed 73°. The condenser is then arranged vertically, and the mixt, is heated under the reflux apparatus, until the b. p. has fallen below 73°. The condenser is then reverted, and the mixt. again distilled until the temp. begins to exceed 73°. The operations are repeated as long as any liquid distils over below 73°. The distillate containing a mol. compd. of alc. and AcOEt, b. about 72° (loc. cit.), is purified in the B. F. P. BRENTON. usual way.

Quinhydrones. WILHELM SIEGMUND. Tech. Hochsch., Vienna. J. prakt. Chem., 83, 553-5.—By the addition of a warm ethereal soln. of the phenol to a warm soln. of p-benzoquinone in petroleum the following quinhydrones have been obtained: 3 C<sub>6</sub>H<sub>4</sub>O<sub>2</sub>, 4 C<sub>6</sub>H<sub>2</sub>(OH)<sub>2</sub>, m. 78°, black needles, from p-benzoquinone (2 mols.) and pyrogallol (1 mol.); 3 C<sub>6</sub>H<sub>4</sub>O<sub>2</sub>, 4 C<sub>6</sub>H<sub>3</sub>(OH)<sub>2</sub>, m. 164°, green needles, from p-benzoquinone (1 mol.) and hydroxyquinol (1 mol.); 3 C<sub>6</sub>H<sub>4</sub>O<sub>2</sub>, 2 C<sub>6</sub>H<sub>3</sub>(OH)<sub>2</sub>, m. 103°, red crystals from p-benzoquinone (4 mols.) and phloroglucinol (1 mol.); C<sub>6</sub>H<sub>4</sub>O<sub>2</sub>, C<sub>10</sub>H<sub>6</sub>(OH)<sub>2</sub>, from p-benzoquinone (1 mol.) and 2,3-dihydroxynaphthalene (1 mol.), separates initially in pale red leaflets, finally in garnet-red needles, both forms m. 78-80°, (vide C. A., 3, 1395).

Synthesis of Glucovanillic Acid and of Gluco-p-hydroxybenzoic Acid. Ferdinand Mauthner. Chem. Inst., Univ. Budapest. J. prakt. Chem., 83, 556-60; vide C. A., 4, 1471.—Methyl tetraacetylglucovanillate,  $C_{22}H_{12}O_{12}$ , m. 144-5°, obtained by shaking a sol. of Me vanillate in dil. NaOH with ethereal  $\beta$ -acetobromoglucose for 26 hrs., is hydrolyzed by aqueous Ba(OH)<sub>2</sub> at the ordinary temp., yielding Tiemann and Reimer's glucovanillic acid. In a similar manner, Me-p-hydroxybenzoate and  $\beta$ -acetobromo-

glucose yield methyl tetraacetylgluco-p-hydroxybenzoate, C<sub>22</sub>H<sub>26</sub>O<sub>13</sub>, m. 159-60°, which is converted into gluco-p-hydroxybenzoic acid, C<sub>12</sub>H<sub>16</sub>O<sub>2</sub>, m. 211-12°, by shaking with 6% Ba(OH)<sub>2</sub> for 24 hrs. at the ordinary temp.

B. F. P. B.

Naphthenic Acids, their Derivatives and Uses. K. V. Kharichkov. Chem. Ztg., 35, 731.—A summary of the author's work on the naphthenic acids.

B. F. P. B.

Preparation of Indigotin as a Laboratory Exercize and as a Lecture Experiment. FRANZ MICHEL. Chem. Ztg., 35, 755-6.—Ten parts of phenylglycine-o-carboxylic acid, or the corresponding amts. of the Na or K salt, are dissolved in 10-12 parts of a soln. of NaOH (1: 4-6), and the soln. evapd. rapidly to dryness on the H<sub>2</sub>O bath. The powdered residue is then added, stirring well meanwhile, to 8-14 parts of paraffin wax (m. circa 60°) heated at 250-70° in a Ni Crucible. The reaction is complete when the fused material becomes strongly yellow. The cold product may then be extracted with hot H<sub>2</sub>O, air being excluded, or after the addition of Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>, or the paraffin may be dissolved away with CHCl2, etc. The products containing indoxyl are then oxidized to indigotin by b. the aq. soln. in the air, a 90% yield of indigotin being obtained. Indirubin may be obtained as follows: the fusion is dissolved in boiling 10% AcOH, the filtrate from the paraffin made strongly alkaline with NH<sub>2</sub>, and the indigotin pptd. by oxidation with a rapid current of air. Extraction with Et<sub>2</sub>O then dissolves the indirubin, indigotin remaining undissolved. For a lecture expt., 0.5-1 g. of the dry mixt. of phenylglycine-o-carboxylic acid and NaOH is heated in a test tube until H.O is no longer evolved, and the fused material has become yellow. After cooling, the mass is b. with 4-5 cc. of H<sub>2</sub>O, the test tube is half filled with H<sub>2</sub>O, and shaken in the air, when indigotin is deposited. B. F. P. B.

from bornylenecarboxylic acid (Bredt, Ann., 348, 206; C. A., 3, 2797). Bornylene-carboxyl chloride,  $C_{10}H_{14}COCl$ ,  $b_{14}$  114-5°, when treated with  $N_{2}H_{4}$ ,  $H_{4}O$  yields the corresponding hydraxide,  $C_{10}H_{15}CONHNH_{2}$ , m. 109-10°, which is converted, according to the method of Curtius, into the axide,  $C_{10}H_{15}CON_{2}$ , and then into  $\beta$ -iminocamphor. On hydrolysis with acids, this yields  $\beta$ -camphor, b. 213.1-13.4°, m. 184-5°, which is identical with epicamphor (m. 165°) recently described by Lankshear and Perkin (Proc. Chem. Soc., 27, 166). On oxidation with HNO<sub>2</sub>,  $\beta$ -camphor yields camphoric acid. Wagner's  $\beta$ -camphor (Chem. Zig., 27, 721) consists mainly of ordinary camphor.

Viscose from Cell Material [Cellulose] and Starch Viscose. H. OST, F. WESTHOFF AND L. GESSNER. Techn. Chem. Lab. Techn. Hochsch., Hannover. Ann., 382, 340–60.—Starch viscose is best prepared by treating starch ( $C_0H_{10}O_0$  1 mol.) with NaOH (2–3 mols.), in 5–20% aq. soln. and  $CS_2$  (20-5 cc. to 10 g. of starch), the mixture is shaken and allowed to react during 24 hrs. The product is then dropped gradually into alc., the viscose collected, dissolved in  $H_2O$  and the treatment with alc. repeated until the product is colorless and liberates no  $CS_2$  when acidified. The substance is dried by kneading it first with alc. then with  $Et_2O$ ; after drying it forms a voluminous material, which may be powdered. Its composition varies, but it is regarded as being essentially a normal Na xanthogenate (NaOC<sub>2</sub>H<sub>2</sub>O<sub>2</sub>OCS<sub>2</sub>Na)<sub>x</sub>, derived from di-Na starch,  $[C_0H_2O_3(ONa)_2]_2$ . Like cellulose viscose, starch viscose, "ripens," i. e., it changes its viscosity with time. At 20°, a specimen showing an initial viscosity of

130 had only a viscosity of 80 and 9 after 96 and 600 hrs., resp., but it showed no sign of coagulation even after many mos. Cellulose viscose, on the other hand, first becomes less viscous, then more so, and coagulates after 4-6 ds., at the ordinary temp, The measurements were made with Engler's viscosimeter. This "ripening" is associated with a hydrolysis, similar in its nature to that which occurs during the purification of the viscose by the method described above. A specimen which had been pptd. thrice and allowed to remain during 12 ds. had the composition C.H.,O.,(ONa)-OCS, Na, but even after 2 mos. no starch was pptd. The fall in viscosity is due to the interaction of the starch and the NaOH and is not caused by the hydrolysis. This was proved by the observation that a viscose, prepared from fresh starch, had the initial viscosity 116, which fell to 28 and 22, after 14 and 21 ds., resp. Fresh viscose prepared from starch which had been treated with aq. NaOH (10%) during 14 and 21 ds., had the viscosity 31 and 27, resp. Starch which has been treated with alkali in this manner, during 21 ds., gives, after purification, the ordinary blue color with I; it does not reduce Fehling's soln. and has  $[a]_b$  195-6°. Similar results were obtained with starch which had been recovered from recently prepared viscose. Soluble starch and dextrins of high mol. wt. give, with NaOH and CS, xanthonates which have a low viscosity even when recently prepared. The inference is drawn, therefore, that the action of NaOH on the starch, as such, or in the form of viscose, consists in a resolution of the complex mol.,  $(C_0H_{10}O_0)n$ , into aggregates of smaller mol. wt., which exhibit the ordinary reactions of starch. Cellulose viscose is prepared best by the use of 15% aq. NaOH. With alc., salts, or acids it behaves like the starch compd., it is pptd. by the first 2 and is decomp. into H.S, CS, and cellulose (or starch) by acids. Cellulose viscose appears to be essentially [C.H.O. (ONa)OCS, Na]m, as suggested by Cross and Bevan. Its stability is greatly increased by the presence of free NaOH; for example, a specimen of viscose containing cellulose (2 g.), free NaOH (6-9 g.) and H<sub>2</sub>O (100 cc.) remained unchanged during 3 mos.; the stability decreases, however, with a larger proportion of NaOH. The process of "ripening" is similar in its nature to that of starch viscose and was tested by corresponding expts. with cellulose and aq. NaOH. A specimen of viscose 5 ds. old, analyzed shortly before coagulation, had the comp. C<sub>24</sub>H<sub>24</sub>O<sub>18</sub>(ONa)OCS<sub>2</sub>Na. These results simply confirm the conclusions of Cross and Bevan, but, whereas, these chemists regarded the "ripening" as including a regeneration of the complex cellulose mol., the present authors consider that it involves a resolution of the mol. The term "hydrate cellulose" has been applied to the cellulose regenerated from viscose and also to cellulose from mercerized and alkali celluloses, etc. Cross and Bevan state that it has the formula (C<sub>2</sub>H<sub>10</sub>O<sub>4</sub>). H<sub>2</sub>O, even after having been dried at 150°. The authors have investigated cellulose recovered from old specimens of alkali cellulose and viscose. The cellulose was dried at 120-5°. Its analyses agreed with the formula, C<sub>6</sub>H<sub>10</sub>O<sub>6</sub>, and gave no evidence that oxidation, or hydrolysis, had taken place. The term "hydrate cellulose" is, therefore, considered to have no significance. The action of alkalies on cellulose, or on starch, is probably very different from that of acids, it may consist only in a depolymerization, which does not involve combination with H.O. nor the conversion into OH of ketonic or lactonic O ats. The formation of dextrins affords examples of such reactions. At 70°, malt extract and starch give a product which, after purification with alc. (40%), consists of erythrodextrins; it has little or no reducing power;  $[a]_0$ 196-8°. At 50°, malt extract gives achroodextrin, which was purified by means of alc. (80%); its reducing power is 15 (maltose = 100);  $[\alpha]_0$  about 190°. The comp. of this dextrin is approx.  $(C_aH_{10}O_b)_7$ .  $H_aO$  and of the erythrodextrins,  $(C_aH_{10}O_b)_7$ . These, J. BISHOP TINGLE. however, are undoubtedly hydrolysis products.

Quinine Alkaloids. XIII. Fluorescence Phenomena of the Quinine Alkaloids.

PAUL RABE AND OSWALD MARSCHALL. Chem. Lab., Univ. Jens. Ann., 382, 360-4 -Filtered ultraviolet light causes the following compds., as solids, to exhibit the fluorescence colors mentioned. Tzvyet's app. was employed: (1) Cinchonine, blue; (2) cinchonidine, pale blue; (3) quinone, intensely blue; (4) quinidine, pale blue; (5) quinine chloride, intensely blue; (6) quinidine chloride, yellowish red; (7) desoxyquinine, reddish blue; (8) desoxyquinidine, sky-blue. The following compds, were examined by diffused daylight, in dil. H2SO4 (1:50) containing 0.1 g. of anhydrous material in 100 cc.; (9) quinine, light blue; (10) quinidine, blue; (11) desoxyquinine, deep violet; (12) desoxyquinidine, violet. The observed fluorescence was always more intense in light from a uviol lamp than in sunlight. The quality and intensity of the fluorescence is greatly dependent on the nature of the solvent, but no regularities could be detected in this connection. When alc. solns, of the above compds, are diluted gradually with H<sub>2</sub>O, an increase in the fluorescence is frequently observed, it is followed by a decrease, as the dilution proceeds. The following substances were examined by means of sunlight, in H<sub>2</sub>SO<sub>4</sub>: (13) quinine, light blue; (14) quinidine blue; (15) quinine and (16) quinidine chlorides, none; (17) cinchonine, blue; (18) cinchonidine, blue; (19) cinchonine and (20) cinchonidine chlorides, none; (21) desoxyquinine, deep violet; (22) desoxyquinidine, violet; quininone, green; quinene, light green; quinic acid and its Et ester, none; p-methoxyquinoline, sky-blue; desoxycinchonine and desoxycinchonidine, bluish violet; cinchene (in uviol light), green; cinchoninone and cinchonic acid, none. Of the compds. mentioned above, those with odd numbers are stereoisomers of the next highest even numbered substance. The influence of acids when used as solvents, is illustrated by the following results: The liquids were 0.1 N and contained 0.1 g. of quinine, in 200 cc. The fluorescence was strong in H<sub>2</sub>SO<sub>4</sub>, HNO<sub>2</sub>, H<sub>2</sub>PO<sub>4</sub>, HF and trichloroacetic acid; medium in tartaric acid and in AcOH; feeble in HI, HBr and in HCl (see following abstrs.).

Quinine Alkaloids. XIV. Scission of Isonitrosoquinotoxine. PAUL RABE AND ERNST MILARCH. Chem. Lab., Univ. Jena. Ann., 382, 365-8; see preceding and following abstrs.—Isonitrosoquinotoxine, in CHCl<sub>2</sub>, when treated with PCl<sub>3</sub>, at o°, gives quinic acid and, in small quantity, mesoquininenitrile. Under similar conditions, isonitrosomethylquinotoxine forms quinic acid and N-methylmeroquinenenitrile.

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Quinine Alkaloids. XV. Partial Synthesis of Cinchonine. PAUL RABE. Chem. Inst., Univ. Jena. Ber., 44, 2088-91; see preceding abstrs.—N-Bromoquinotoxine formula (I) below, is prepared, at the ordinary temp., by adding gradually, while

vigorously stirring, Br (32 g.), in 6% aq. NaOH (400 g.) to quinotoxine (58 g.), in 1 N aq. HCl (200 cc.) and Et<sub>2</sub>O (500 cc). After 10 mins. the Et<sub>2</sub>O soln. is removed and dried. Long, colorless prisms from Et<sub>2</sub>O, or alc., m. 153°. Yield, about 54%. It is neutral to litmus and does not react with MeI. The conversion of (I) into cinchoninone (II) is best effected by adding to the b. soln. of (I) (10 g.), in alc. (250 cc.), cold,

alc. EtONa soln. (30 cc.), containing Na (1.5 g.). After being allowed to cool spontane-

ously, the liquid is acidified with dil. HCl, steam distilled until free from alc. and the residue extracted with  $Et_2O$ . The reduction of (II) to cinchonine (III) has been described previously (C. A., 2, 1146).

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Condensation of a Substituted Formamide to a Derivative of Aminomalonyldiamide HERMAN DECKER AND PAUL BECKER. Org. Chem. Lab. Techn. Hochsch., Hannover. Ann., 382, 369-77.—Treatment of formylphenylethylamine, PhCHC.H. NHCHO, with  $P_4O_{100}$  by Bischler and Napieralski's method (Ber., 26, 1903 (1893)), gives a yellow resin, from which b. alc. (95%) extracts \(\beta\)-phenylethylaminomalonyldiphenylethylamide phosphate; slender, colorless needles, m. 176-8°. Picrate, C2,H2,O2,N2,C4H2,N2O2, from the preceding compd., in alc.; crystallin, m. 192°. Hydrochloride, C27H21O2N2.HCl, prepared like the picrate; white plates from alc. (80%) containing HCl, m. 184-6°. Chloroplatinate, (C<sub>27</sub>H<sub>21</sub>O<sub>2</sub>N<sub>3</sub>)<sub>2</sub>.H<sub>2</sub>PtCl<sub>2</sub>, yellowish red, lustrous plates from alc., darkens about 235°, m. 254-5°. \$\textit{\theta-Phenylethylaminomalonyldiphenylethylamide}, (PhCH\_cH\_s-NHCO)2CHNHCH2CH2Ph, from the picrate and NH2, in presence of C2H2; slender, white needles from alc., m. 85°; mol. wt., in freezing CaHe, 394-401. At 165°, during 8 hrs., with aq. HCl (20%), it is hydrolyzed to CO<sub>2</sub>,  $\beta$ -phenylethylamine, PhCH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub> and  $\beta$ -phenylethylaminoacetic acid, PhCH<sub>2</sub>CH<sub>2</sub>NHCH<sub>2</sub>CO<sub>2</sub>H, which are sepd. either by the greater solubility of the acetic acid hydrochloride in H.O, or alc., or by steam distillation of the free amines, in this case the  $\beta$ -phenylethylamine passes over. Alk. KOH also hydrolyzes the malonylamide, the reaction being completed after b. during 20 hrs. The \(\theta\)-phenylethylaminoacetic acid was likewise prepared by heating \(\theta\)-phenylethylamine, chloroacetic acid and AcONa, during 3 hrs., on a H,O bath; slender, white needles from alc. (80%), m. 274-6°. Hydrochloride, C1. H12O2N.HCl, silvery white plates from dil. HCl, m. 243-4° (gas evolution), when rapidly heated. These results explain why 3,4-dihydroisoquinoline could not be prepared by the condensation of formylphenylethylamine, although acetylphenylethylamine gives an isoquinoline derivative without difficulty. J. BISHOP TINGLE.

Constitution of Camphene. II. Camphene Hydrochloride and Camphene Hydrate. OSSIAN ASCHAN. Chem. Lab., Univ. Helsingfors. Ann., 383, 1-38; see C. A., 4, 3200.—Unless otherwise stated, the camphene employed in the expts. described below was prepared by the action of bases on the hydrochloride of rectified American turpentin oil; it had the following physical constants: b.  $159-60^{\circ}$ ; m.  $40.5-1.5^{\circ}$ ;  $[\alpha]_{20}^{20}$ 1.15°; in  $C_nH_{av}$  [ $\alpha$ ] $^{20}_{D}$  12.85°. In Et<sub>2</sub>O, with dry HCl, it gave a hydrochloride, m. 137-40°; after recrystallization from MeOH, m. 142-3°. Yield, 102.6% of the camphene. It appears to dissociate into its constituents at 175-8°. After treatment of the hydrochloride (500 g.) with C<sub>8</sub>H<sub>8</sub> (40 g.), KOH (160 g.) and H<sub>2</sub>O (6 l.), at 60° and then at 80-90°, during 12 hrs. at each temp., a product consisting of camphene and its hydrate was obtained; the highest m. p. shown by the latter was 150-1°. The hydrate differs from borneol and isoborneol in the following respects: (1) It is pptd. as a viscid oil on adding H<sub>2</sub>O to its soln. in alc., or MeOH. (2) It gives a milky turbidity if 0.1 g. is treated with 2 cc. of HNO<sub>2</sub> (d. 1.4); when heated the liquid becomes orange-yellow then evolves red fumes; after dilution with H<sub>2</sub>O and neutralization with NaOH the color is orange-yellow. Under similar conditions, natural l-borneol gives a clear soln. which evolves red fumes at a lower temp, than in the case of camphene hydrate and becomes yellow. The color is discharged by dilution and addition of NaOH. Synthetic isoborneol reacts like camphene hydrate, but the colors are feebler and no rapid evolution of red fumes takes place. Camphene gives a clear soln., which becomes reddish yellow when heated. The evolution of red fumes is often quite sudden. After adding H<sub>2</sub>O and NaOH the color is strongly reddish yellow. It is evident, from these results, that the first action of HNO, consists in a conversion of camphene hydrate into camphene. B., glacial AcOH (2.5 pts.) also changes camphene hydrate into

camphene, a little isobornyl acetate being produced simultaneously, probably from the camphene. Ac<sub>2</sub>O + AcONa act in a similar manner. Dry HCl converted camphene hydrate, in C<sub>2</sub>H<sub>4</sub>, into camphene hydrochloride. After 3 hrs., at 60-70°, camphene hydrate (30 g.) is transformed almost completely into isobornyl acetate by H<sub>2</sub>O (0.5 cc.), conc. H<sub>2</sub>SO<sub>4</sub> (4 g.) and 98.7% AcOH (80 g.). Oil from Siberian pine needles was fractionated and the portion containing the pinene-camphene was oxidized, so as to remove the pinene. The resulting camphene, b. 159-9.5°; m. 47-8°; den 0.8548;  $[\alpha]_{\rm D}^{60}$  -76.33°;  $[\alpha]_{\rm D}^{60}$  -89.29°;  $\pi$  1.45952; mol. ref. 43.54. Its hydrochloride, prepared in MeOH, after recrystallization from C<sub>2</sub>H<sub>4</sub>, m. 149-50°; in C<sub>2</sub>H<sub>4</sub>, [α]<sub>b</sub> 8.66°; [ $\alpha$ ] 47.33°. With aq. KOH (3%), at 80°, in the manner described above, it gave camphene hydrate having the following constants: in  $C_pH_{q_p}[\alpha]_{p_p}^{20}$  —0.12°;  $[\alpha]_{p_p}^{20}$  —1.35°; mol. ref. 44.45. B. with glacial AcOH + AcONa converted it into camphene, bree  $161-3^{\circ}$ ; m.  $49.5-51^{\circ}$ ; in  $C_6H_{40}$  [ $\alpha$ ] $^{20}_B$  — $6.72^{\circ}$ ; [ $\alpha$ ] $^{20}_B$  — $76.04^{\circ}$ . This series of reactions produced, therefore, no change in the optical properties of the camphene. The results of the experiments described above and of others recorded in the original paper, may be summarized as follows: (1) Camphene,  $[\alpha]_D$  12.85°  $\longrightarrow$  (2) camphene hydrochloride,  $[\alpha]_{\mathbb{D}}$  —6.40°, the  $\beta$ -isomer  $\longrightarrow$  (3) camphene,  $[\alpha]_{\mathbb{D}}$  10.97°. The  $\alpha$ -isomer of (2)  $\longrightarrow$  (4) camphene hydrate,  $[\alpha]_b - 2.89^\circ$  which  $\longrightarrow$  (5) camphene hydrochloride,  $[\alpha]_{\mathbf{b}}$  —6.49°; but (4), when dehydrated  $\longrightarrow$  (6) camphene,  $[\alpha]_{\mathbf{b}}$ 12.63°. These changes to and from camphene, its hydrate and its hydrochloride do not affect its optical properties and therefore, presumably, involve no rearrangement of the C nucleus. In spite of the fact that camphenes (3) and (6) were formed from isomeric hydrochlorides, they are essentially identical with (1) and indicate that camphene is homogeneous. The isomeric hydrochlorides have not been separated, their relationship to each other and to camphene formula (I) below, is shown by (II) and

(III). Measurements have been made of the progress of the hydrolysis, at 50°, of pinene and camphene hydrochlorides and of bornyl and isobornyl chlorides. The expts. were made with 0.5 N alc. KOH, in excess. The results are reproduced in the form of curves; they show that isobornyl chloride contains 60-70% of a very reactive compd. (camphene β-hydrochloride) and about 25% of camphene α-hydrochloride, which reacts much more slowly; the remainder consists of a third isomer, which is still more stable. Bornyl chloride and camphene hydrochloride contain only about 50% of the  $\beta$ -isomer and 8–10% of the third stable modification, which was not hydrolyzed even after 30 hrs., with alkali in excess. Pinene hydrochloride, after repeated recrystallization, contains about 5% of the  $\alpha$ - and  $\beta$ -isomers. Directions are given for the preparation of homogeneous specimens of bornyl and isobornyl chlorides and of pinene hydrochloride. Camphene hydrate exhibits the characteristic properties of a tertiary alc. Pinene hydrochloride, from Greek turpentine oil, after purification, had the following properties: m., 128-30°; freezing p., 127.5°; in  $C_6H_{\bullet}$  [ $\alpha$ ] $^{20}_D$  28.88°. When treated with milk of lime and CaHe, at 135°, during 8 hrs., then at 150°, during 15 hrs. it gave a hydrate, which, after sublimation, m. 149-50°; in  $C_0H_0$ ,  $[\alpha]_D^{20}$  -21.79°. It follows, therefore, that "camphene hydrate" may exhibit a considerable degree of optical activity. With glacial AcOH + AcONa, in the manner described above, it formed "camphene" having  $[\alpha]_{p}^{20}$  85.68°. In the author's opinion this shows "that the tert. terpene alc. from pinene hydrochloride is identical with the camphene hydrate from camphene hydrochloride." The grounds on which this conclusion is based are

not clearly apparent. The values of  $[\alpha]_D$  for the hydrates of different origin vary widely, as recorded above and yet, as shown above, the author relies on the constancy of  $[\alpha]_D$  to demonstrate the homogeneity of camphene. The frequent presence of Cl, in specimens of the above terpenes, which have been prepared from the hydrochlorides, is ascribed to the presence of the third, stable isomeric hydrochloride. It is not decomp. by PhNH<sub>2</sub>, at the ordinary temp., nor by dil., aq. KOH, at 80–90°, but when heated with PhNH<sub>2</sub> reaction takes place (see following abstr.).

J. BISHOP TINGLE.

Constitution of Camphene. III. Homogeneity of Camphene of Different Origin. OSSIAN ASCHAN. Chem. Lab., Univ. Helsingfors. Ann., 383, 39-51; see preceding and following abstrs.—The varieties of camphene mentioned below were investigated, in the manner described. (1) Com. camphene, prepared by the action of bases on pinene hydrochloride (from American turpentin oil). After careful fractional distillation it b. 159.5-60°; m. 41-2°; in  $C_0H_0$ ,  $[\alpha]_D^{20}$  1.87°;  $[\alpha]_D^{20}$  10.7°. (2) Camphene from isoborneol. Isobornyl chloride was distilled with PhNH, the product was optically inactive; b. 158-9°; m. 49-51°. (3) Camphene from l-borneol. This was prepared in the same manner as (2); b.  $158.5-9.5^{\circ}$ ; m.  $49.5-50.5^{\circ}$ ; in  $C_0H_{av}$  [ $\alpha$ ]<sub>b</sub>  $-63.7^{\circ}$ . (4) Camphene from Kahlbaum. It was identical with that used by Harries and Palmen (C. A., 4, 2488) and was optically inactive; b. 158-9°; m. 48-9°. (5) Camphene from Siberian pine needle oil, b. 155-60°. It was prepared by the action of H<sub>2</sub>SO<sub>4</sub> (55%), at the ordinary temp. and was optically inactive; b. 159-60°; m. 38-9°. (6) Camphene from (1). At the ordinary temp., a soln. of (1) (2 kg.), in hot MeOH (3 kg.), deposited 918 g. It was dried over CaCl, while molten and distilled over Na; b. 159.5-60°; m. 46-7°; in  $C_0H_0$  [ $\alpha$ ] $^{20}_D$  1.63°; [ $\alpha$ ] $^{20}_D$  9.37°. (7) Camphene from pinene hydrochloride. This was obtained from Greek turpentin oil, which was strongly d-rotatory; b. 158-9°; m. 43-4°;  $[\alpha]_0$  59.27°. It was not identical with the camphene of similar origin described in the latter part of the preceding abstr. The above specimens of camphene were oxidized as follows: the camphene (25 g.) was mixed with C<sub>2</sub>H<sub>4</sub> (5 g.), H<sub>2</sub>O (500 cc.) and KOH (7.5 g.) and to this soln. was added gradually, KMnO<sub>4</sub> (64 g.), in H<sub>2</sub>O (3.2 l.), at the ordinary temp. Eventually alc. was added, if necessary to decolorize the liquid. The product was filtered, the solid steam distilled, the distillate extracted with Et,O and, after its evaporation, the residue which had dissolved in Et<sub>2</sub>O was heated at 170°. The portion not volatil at that temp. was camphenilone. The first filtrate and also the distillate which had been extracted with Et<sub>2</sub>O, were combined and extracted 4 times with Et<sub>2</sub>O (total 400 cc.) this removed camphenegiycol. The aq. liquid was concentrated and excess of Na<sub>2</sub>CO<sub>2</sub> added to ppt. Na camphenilate. The filtrate was conc. and allowed to remain at oo, it deposited sometimes a small quantity of a sodium salt, probably of a monobasic acid; lustrous, quadratic plates, m. about 138°. Yield, very small. From the filtrate, camphenic acid was pptd. by means of conc. HCl and, after its removal, Et,O extracted another acid or acids in small quantity. For the details of the further separation and purification of the compds, mentioned above the original paper must be consulted. The following numbers give the quantities of the various oxidation products, in % of the varieties of camphene described above: A - camphenilone; B - campheneglycol; C - camphenilic acid; D - the Na salt mentioned above; E - camphenic acid; F - soluble acids mentioned above. (1) A, 1.20; B, 8.0; C, 5.8; D, 1.4; E, 64.0; F, 5.0. (2) A, 1.68; B, 6.7; C, 5.6; D, o; E, 65.0; F, 6.5. (3) A, 1.60; B, 6.6; C, 6.8; D, o; E, 71.6; F, 4.6. (4) A, traces; B, 10.4; C, 5.4; D, 0; E, 70.4; F, 6.8. (5) A, 2.6; B, 5.4; C, 6.3; D, 1.2; E, 81.7; F, 9.1. (6) A, 1.40; B, 8.4; C, 6.8; D, 0.3; E, 64.2; F, 5.7. (7) A, traces; B, 10.4; C, 6.9; D, traces; E, 81.1; F, 6.5. These results indicate that, apart from optical isomers, camphene is homogeneous. The ratio C: E is about 1: 10, i. e., the oxidation products derived from a semicyclic camphene, are much smaller in quantity than those derived from a camphene containing the ethylene linkage in the nucleus: Assuming that camphene is homogeneous, C (and probably also A and B) would be formed much more easily by rearrangement from the ethylene form of camphene, than would E from the methylene form. The former involves the transformation of a 6-membered into a 5-membered ring and such changes are known to take place frequently and without difficulty. The acceptance of the methylene formula for camphene involves the assumption that a ring of 5 members has become converted into a closed chain of 6 members and that this has taken place with extreme ease. because the product (E) of the change, constitutes by far the larger portion of the oxidized material. It is, however, well-known that the formation of 6-membered cycloids from those of 5 members can be accomplished only with great difficulty. These results lead to the conclusion that camphene either contains an ethylene linkage in the nucleus, or that it is a tricycloid with a trimethylene ring, which changes readily into a nucleus containing an ethylene linkage. . BISHOP TINGLE.

Constitution of Camphene. IV. Stereoisomeric Camphenic Acids. Ossian ASCHAN.. Chem. Lab., Univ. Helsingfors. Ann., 383, 52, 68; see preceding abstrs. and C. A., 4, 3200.—The camphenic acid employed, m. 135.5-6.5°. When heated at 180°, during 12 hrs., with glacial AcOH (2 pts.) and 38% HCl (2 pts.), it gives a mixture of cis- and trans-acids, which were separated by treatment with abs. alc. and EtONa. The less sol. Na salt (about 80%) consists of unchanged cis-acid. The remainder is dl-transcamphenic acid, m. 122-3°, not 120.5°, as stated previously. The cis-acid was purified by means of H<sub>2</sub>O and the isomer by recrystallization from dil. AcOH. dl-Transcamphendiamide, CaH14(CONH4)2, pointed, rhombohedral plates from glacial AcOH, m. 231-2°. The cis-diamide m. 225° not 222°. A mixture of both m. 215°. Trans-dl-anilide, prisms from glacial AcOH + H.O, m. 165°. The cis-dianilide m. 212°, not 210°, a mixture of both m. 160-200°. In glacial AcOH + HCl, "Zn wool" reduces cis-α-bromocamphenic acid to a mixture of cis- and wassdl-camphenic acids which were separated in the manner described above. Some trans-dl-acid is also formed by distilling the cis-dl-acid (5 mm.). When distilled under the ordinary pressure, in CO, most of the cis-acid decomp. The portion of the product volatil with steam consists of an unsaturated acid, CoH14O3; oil, ba 132.5-3.5°. It decolorizes KMnO<sub>4</sub> instantly and does not react with semicarbazine. Ammonium salt, quadratic plates. Barium salt, aggre of rods. Calcium sa t, sparingly sol. Silver salt, CoH13O2Ag, white and curdy. 1. ...cid is oxidized by HNO3 to a mixture of crystallin acids. The portion of the distillation product not volatil with steam (see above) consists of an acid, C10H14O1; aggregates of rods, or thin, lustrous plates from H<sub>2</sub>O, m. 134°. When mixed with the parent cis-acid, m. 110-7°. It reacts with semicarbazine, but the resulting material was not homogeneous. Calcium salt, sparingly sol. Barium salt, readily soluble. Sodium salt. Adipic acid, when distilled, decomp. into cyclopentanone, an unsaturated, oily acid, probably CH2: CHCH2-CH<sub>2</sub>CO<sub>2</sub>H and a saturated, oily acid. The solubility, at 19.5°, of trans-dl-camphenic acid is stated to be 0.3122 (presumably g.) in 100 g. of soln., but the nature of the solvent is not stated. The following camphenic acids are known at present: Cis-dl-, m. 135.5-6.5°; cis-d-, m. 143.5-4.5°; cis-l-, m. 143.5-4.5°; trans-dl-, m. 122-3°.

J. BISHOP TINGLE.

New Synthetic Glucosides. EMIL FISCHER AND BURCKHARDT HELFERICH. Chem. Inst., Univ. Berlin. Ann., 383, 68-91; see C. A., 4, 3234.—Tetraacetyl- $\beta$ -benzyl-d-glucoside, PhCH<sub>2</sub>C<sub>6</sub>H<sub>7</sub>O<sub>6</sub>Ac<sub>4</sub>, is prepared by shaking acetobromoglucose, with dry Et<sub>2</sub>O, benzyl alc. and dry Ag<sub>2</sub>O; long, white, silky lustrous needles from alc. (50%), m. 96-101° (cor.); in alc.,  $[\alpha]_{22}^{12}$ —49.51° (±0.6°). Yield, 72%. The compd. does

not reduce b. Fehling's soln. It is hydrolyzed by prolonged warming with dil. HCl. β-Benzyl-d-glucoside, PhCH<sub>2</sub>OC<sub>2</sub>H<sub>11</sub>O<sub>A</sub>, from the preceding compd. and aq. Ba(OH)<sub>2</sub>, at the ordinary temp., during 16 hrs.; small, soft needles from Et<sub>2</sub>O, m. 123-5° (cor.); in  $H_2O$ ,  $[\alpha]_0^{20}$  -55.76° (±0.4°). From AcOEt needles containing  $H_2O$  are deposited. Yield, 97%. The compd. has a strongly bitter taste, but no odor, it does not reduce Fehling's soln. and it is rapidly hydrolyzed by warm, dil. HCl, or by emulsin. The glucoside may also be prepared from the tetraacetyl compd. by hydrolysis with aq. NH<sub>a</sub>. Unless otherwise stated, the compds. described below were prepared by the same methods as those employed in the case of the preceding substances, Ba(OH), being used for the hydrolysis of the Ac derivatives. Tetraacetyl-β-cyclokexanol-d-glucoside, C<sub>2</sub>H<sub>11</sub>C<sub>2</sub>H<sub>2</sub>O<sub>2</sub>Ac<sub>4</sub>, from cyclohexanol; long, silky lustrous needles from alc. (25%), m. 120-1° (cor.); in alc.,  $[\alpha]_{D}^{21}$  -29.74° (±0.5°). Yield, 65%.  $\beta$ -Cyclohexanol-d-glucoside, CaH110CaH110a, needles aggregated into thick crusts from AcOEt, m. 133-7° (cor.), previously softening; in H<sub>2</sub>O,  $[\alpha_{D}^{20}]$  —41.43° (±0.2°). Yield, 93%. The glucoside is hydrolyzed by emulsin. Tetraacetyl-β-geraniol-d-glucoside, C<sub>10</sub>H<sub>1</sub>,C<sub>6</sub>H<sub>7</sub>O<sub>6</sub>Ac<sub>4</sub>, from geraniol; crystals from dil. alc., m. 29-30°; in alc.,  $[\alpha]_D^{22}$  —25.17° (±0.25°); in contact with H<sub>2</sub>O, m. about 20°. Yield, 58%. β-Geraniol-d-glucoside, C<sub>10</sub>H<sub>17</sub>O-C<sub>2</sub>H<sub>11</sub>O<sub>2</sub>, long, slender needles with 1 H<sub>2</sub>O from AcOEt, m. about 58°; in H<sub>2</sub>O,  $[\alpha]_{\rm p}^{27}$  -37.25° (±0.2°). It has a very bitter taste, is rather hygroscopic and is hydrolyzed by emulsin. Yield, 82%. Tetraacetyl-β-cetyl-d-glucoside, C16H2OC6H2O6AC4, from cetyl alc.; silky lustrous needles from MeOH, m. 71-3° (cor.), previously softening; in alc.,  $[\alpha]_{20}^{20}$  —20.19° (±0.6°). Yield, 33%. It is hydrolyzed by b., glacial AcOH, in presence of a little conc. H<sub>2</sub>SO<sub>4</sub>, but not by dil. HCl, or H<sub>2</sub>SO<sub>4</sub>. β-Cetyl-dglucoside, C12H210C2H11O4, colorless, lustrous, dendritic needles from Et2O, softens about 78°, begins to m. 110°, m. completely about 145°; in alc.,  $[\alpha]_D^{24}$  —22.02° (±0.3°). It is tasteless and is not hydrolyzed by emulsin, nor by dil. mineral acids, doubtless because it is insol. in H<sub>2</sub>O. Yield, 66% of the Ac compd. Ethyl tetraacetyl-β-d-glucosidoglycolate, Ac,CaH,O,OCH,CO2Et, white needles from alc., m. 83-4° (cor.), previously softening; in alc.,  $[\alpha]_D^{23}$  —40.21° (±0.5°). Yield, 60%.  $\beta$ -d-Glucosidoglycolic acid, C.H., O.OCH, CO.H., aggregates of plates from MeOH + Et.O, m. 165-7° (cor.); in  $H_2O$ ,  $[\alpha]_D^{21}$  -44.11° (±0.2°). Yield, 71%. The compd. has an acid taste, resembling that of malic acid; it is hydrolyzed by warm, dil. HCl, but not by emulsin. Calcium salt, Ca(C<sub>2</sub>H<sub>12</sub>O<sub>2</sub>)<sub>2</sub>, amorphous and vitreous. Sodium salt, prepared in MeOH; spherical aggregates of microscopic plates from MeOH. Its soln. is neutral. Zinc, barium, lead and mercury salts, amorphous. All the salts are readily sol. in H2O. Amide, from the ester and dry NH, in MeOH, at the temp. of a freezing mixture; small, hexagonal prisms from abs. alc., softens about 162°, m. 167° (cor.); in H<sub>2</sub>O,  $[\alpha]_{1}^{18}$  -43.24° (±0.2°). Yield, 82%. The amide is sweet, with a bitter after-taste and it is readily hydrolyzed by b., dil. HCl, or by emulsin. When b. during 4 hrs., with Ac<sub>2</sub>O, it is converted into a compound, C<sub>18</sub>H<sub>28</sub>O<sub>12</sub>N, which may possibly be pentaacetylglucosidoglycolamide; white needles from MeOH, m. 146-9° (cor.). about 41%. In MeOH, NH, regenerates the amide. Pentabenzoyl-d-glucose, in CHCl<sub>2</sub>, has  $[\alpha]_D^{19}$  25.37° (±0.2°). When treated with HBr, in glacial AcOH, at 18°, during 1 hr., it gives β-benzobromo-d-glucose, BrC<sub>8</sub>H<sub>7</sub>O<sub>4</sub>Bz<sub>4</sub>; slender, white needles from amyl alc., m. 125-8° (cor.); in toluene,  $[\alpha]_D^{20}$  144.7° (±0.3°). Yield, 100% of the benzoylglucose. With MeOH and Ag<sub>2</sub>O, in the manner described above, it forms tetrabenzoyl-β-methyl-d-glucoside, MeC<sub>2</sub>H<sub>7</sub>O<sub>6</sub>Bz<sub>4</sub>; white needles from MeOH, m. 160-2° (cor.); in CHCl<sub>2</sub>,  $[\alpha]_D^{19}$  30.79° (±0.2°). Yield, 69%. When shaken with a soln. of Na, in ordinary alc., it is hydrolyzed to  $\beta$ -methylglucoside, which was fully identified. J. BISHOP TINGLE.

Theory of Halochromic Phenomena. II. P. PFEIFFER. Chem. Lab., Univ.

Zurich. Ann., 383, 92-155; see C. A., 5, 459; A. Werner, C. A., 5, 2453; E. Fischer, C. A., 5, 3282.—The author again discusses at great length, his theory of the cause of halochromic phenomena. The Walden inversion is also considered and a theory of it is advanced which is essentially identical with that of E. Fischer and A. Werner. For the sake of brevity, in the stannic halide additive compds. described below, the organic constituent is indicated by R, which represents 1 mol. of the resp. aldehyde or ketone. Disalicylic aldehyde stannic chloride, R.SnCl., was prepared from the constituents either without a solvent, or by b. in C.H.; deep yellow, cryst. powder, m. about 152°. The remaining compds. of this class, described below, were prepared in C.H., unless otherwise stated. Disalicylic aldehyde stannic bromide, R.SnBr., prepared like the preceding compd.; small, deep yellow crystals, m. 100°. When exposed to air it forms a hydrate with 2 H<sub>2</sub>O; colorless. Over P<sub>4</sub>O<sub>10</sub> the parent compd. is regenerated. Salicylic aldehyde hydrobromide, R.HBr, from dry HBr and the well cooled aldehyde; unstable, yellow, prismatic crystals. Bis-o-methoxybenzaldehyde stannic chloride, R.SnCl4; yellow, cryst. powder, m. 180-1°. Bis-o-methoxybenzaldehyde stannic bromide, R.SnBr., yellow, cryst. powder, m. 131°. Acetylsalicylic aldehyde stannic chloride, RSnCl., gray, cryst. powder. It quickly liquefies in air and was not prepared in presence of CoHo. Bis-m-hydroxybensaldehyde stannic chloride, R.SnClo yellow, crystallin and stable. Bis-m-kydroxybensaldekyde stannic bromide, R.SnBr., yellow crystals, decomp. without m. when heated. Bis-m-methoxybensoldehyde stannic chloride, R.SnCl., yellow, crystallin powder, decomp. in air. Dipiperonaldehyde stannic chloride, R.SnCl., pale yellow, crystallin powder, darkens about 130°, chars completely about 190°. Dipiperonaldehyde stannic bromide, R.SnBr., pale yellow, crystallin powder, m. 150°. Bis-p-dimethylaminobenzaldehyde stannic chloride, R.SnCl., prepared in abs. Et.O; canary-colored, decomp. without m. when heated. Bis-p-dimethylaminobenzaldehyde stannic bromide, R.SnBr., prepared in abs. Et.O; canarycolored, decomp. without m. when heated. Bis-o-nitrobenzaldehyde stannic chloride, R.SnCl., colorless, prismatic needles, decomp. in air, m. 80-120°. Bis-m-nitrobensaldehyde stannic chloride, R. SnCl., colorless, crystallin powder, decomp. without m. Bis-pnitrobenzaldehyde stannic chloride, R.SnCl., light yellow, transparent, prismatic needles, which soon change to a pale yellow powder containing 0.5 C<sub>n</sub>H<sub>a</sub>; m. about 101°. Bism-hydroxyacetophenone stannic chloride, R.SnCl., pale yellow, unstable, crystallin powder, m. 99°. Bis-p-hydroxyacetophenone stannic chloride, R<sub>2</sub>SnCl<sub>4</sub>, colorless, crystallin powder, becomes deep red about 150°, m. about 190°. Diphorone stannic chloride, R.SnCl., prepared in abs. CHCl.; colorless, transparent crystals, m. about 142° (gas evolution). Bisdimethylpyrone stannic chloride, R.SnCl., colorless, crystallin crusts, m. 232-5° (decomp.). Gomberg and Kyriakides' dixanthone stannic chloride, R<sub>2</sub>SnCl<sub>4</sub>, has also been prepared, m. 245°. Dibenzophenone stannic chloride, R<sub>2</sub>SnCl<sub>4</sub>, prepared in abs. CHCl<sub>2</sub>; colorless, crystallin and unstable. Bisbenzalacetophenone stannic chloride, R.SnCl., aggregates of deep yellow crystals. Bisdibenzalacetophenone stannic chloride, R.St Cla, orange-yellow powder with 1 C.H., which is slowly evolved; when free from solvate the color is pure orange, m. 188° (decomp.). Biscinnamylideneacetophenone stannic chloride, R.SnCl., lustrous, bordeaux-red crystals, which are relatively stable in air, when powdered it is orange-colored, m. about 160° (decomp.). Bisdicinnamylideneacetone stannic chloride, R.SnCl., lustrous, black powder, decomp. below 100°. Its color in b. C<sub>2</sub>H<sub>4</sub> is deep orange-red, in b. alc., brownish yellow. Bisbenzoylpiperidine stannic chloride, R.SnCl., prepared in CHCl.; colorless, crystallin crusts, liquesies in air, m. about 213°. Biscinnamoylpiperidine stannic chloride, R.SnCl., small, colorless, lustrous plates, m. 221°. Dipiperine stannic chloride, R.SnCl. deep yellow, crystallin crusts, decomp. and darkens when heated. Dipiperine stannic bromide, R.SnBr., deep yellow and crystallin, m. and darkens 183°. Piperine hexachlorostannate, RaHaSnCla, from piperine, SnCla and abs. alc. saturated with HCl;

compact, yellow crystals from alc. containing HCl, when heated it becomes orange-colored, then blackens and softens. Piperine hexabromostannate, R<sub>2</sub>.H<sub>2</sub>SnBr<sub>6</sub>, is prepared in a similar manner to the preceding compd., in abs. alc., or abs. MeOH; compact, deep yellow crystals, m. and darkens 182-4°. Piperine hydrochloride, R.HCl, prepared in anhydrous C<sub>6</sub>H<sub>6</sub>; canary-colored and crystallin. Piperine dihydrochloride, R.2HCl, from powdered piperine and dry HCl; orange-colored powder, decomp. in air. Piperine hydrobromide, R.HBr, prepared in anhydrous C<sub>6</sub>H<sub>6</sub>, or in Et<sub>6</sub>O; canary-colored, cryst. powder, m. about 170°. The only product which could be obtained from SnCl<sub>6</sub> and triphenylmethyl chloride was Kehrmann's compd., RSnCl<sub>6</sub>.

J. BISHOP TINGLE.

Preparation of Isoprene from Terpene Hydrocarbons. H. STAUDINGER AND H. W. KLEVER. Chem. Inst. Techn. Hochsch., Karlsruhe. Ber., 44, 2212-5.—Considerable quantities of isoprene are formed by passing the vapor of limonene, or dipentene, diluted with N, over an electrically heated Pt spiral. For laboratory purposes it is more convenient to omit the N and use the hydrocarbon vapor under 20-30 mm. Yield, of isoprene, about 60%. A larger yield is obtained if the vapor be under 2-3 mm. In addition to isoprene there are formed in small quantity trimethylethylene, gaseous hydrocarbons of the C<sub>2</sub>H<sub>4</sub> and C<sub>2</sub>H<sub>2</sub> series and liquid hydrocarbons, b. 100-50°. Hardly any higher b. compds. are produced. The yield of isoprene from turpentine oil vapor, under the conditions described above, is very small and from camphene, or pinene little or no isoprene is formed. It is suggested that this method of experimentation may be employed to differentiate certain of the terpenes (see following abstr.).

J. BISHOP TINGLE.

Decomposition of Terpene Compounds by means of Glowing Metallic Wires. C. HARRIES AND KURT GOTTLÖB. Chem. Inst., Univ. Kiel and Prague, Visocan. 383, 228-9; see 2 preceding abstrs.—The terpenes mentioned below were decomp. in an apparatus consisting of a round-bottomed flask, carrying a condenser, through which H<sub>2</sub>O at any desired temp., say 50°, may be passed. The upper end of the condenser is connected by means of a suitable T-tube to a separatory funnel and also to a second condenser. In the upper part of the flask, just below the neck, is suspended, by means of thick Cu wires, a coil of Pt wire. It resembles the coil of a Ta lamp and contains 120 cm. of wire that can be heated to medium redness, by a current of about 5 amperes, at 220 volts. The Cu wires pass through the stopper of the flask and are suitably connected with the current supply. The terpene is placed in the flask and is b., the vapor coming into contact with the Pt is decomp. The products which are volatil at 50° pass through the first condenser, but are liquefied by the second. Unchanged terpene flows back to the flask. The yield of isoprene, from d- and l-pinene was about 1%, from com. carvene, 30-50%, depending on the limonene content. In addition to the isoprene, gaseous compds. are formed and there remain in the flask thick oils of high b. p., which do not decolorize Br. The isoprene is purified by careful rectifica-J. BISHOP TINGLE.

Nature of Phenolic Aldehydes. I. Reactivity of the Aldehyde Group in Phenolic Aldehydes. H. PAULY AND RICHARD FRH. V. BUTTLAR. Chem. Inst., Univ. Würzburg. Ann., 383, 230-88.—The 8 reactions mentioned below proceed readily with ethers of phenolic aldehydes, yields being 75% or more, but if the phenolic aldehyde contains 1 or more OH groups, or if the reactions are applied to the cyclocarbonates of dihydroxybenzaldehyde, the yields are usually either much reduced, or no reaction at all takes place. The reactions in question are: (1) The production of alc. and acid, by means of KOH. (2) The benzoin condensation, by means of KCN. (3) Formation of acetals, from alc. + HCl. (4) Formation of acetals, from orthoformic esters. (5) Production of hydroxamic acids (Angeli). (6) Grignard's reaction. (7) Condensa-

tion with pyruvic acid and  $\beta$ -naphthylamine. (8) Reaction with fuchsine-SO<sub>2</sub>. This failure of phenolic aldehydes to react is not dependent on the position of the OH in the benzene nucleus, nor on the degree of acidity of the phenolic group or groups and it cannot be ascribed to steric influences. The authors conclude that the phenolic O at. is the cause of this inhibition of the reactivity of the CHO group and they discuss and formulate the facts at great length in terms of J. Stark's valence theory (C. A., 2, 2644). It is difficult to understand this conclusion regarding the O at. in view of the facts that the cyclic carbonates fail to react, whereas the phenolic ethers react reactily in every case (see above). When treated in alc., with a rapid current of HCl, the following derivs. of BzH give the colors mentioned: 2-hydroxy-, light red; 3-hydroxy-, bluish fluorescence; 4-hydroxy-, wine-red; 1,3-dihydroxy-, salmon-red; 2,4 -dihydroxy-, very intense fuchsine-red; 2,5-dihydroxy-, violet-red; 3,4-dihydroxy-, dark violetred; its cyclocarbonate and its Me 3-carbonate, wine-red; 4-methoxy-, carmin-red; 3,4-methyleneoxy-, wine-red; 2-hydroxy-3-methoxy-, salmon-red; 3-hydroxy-4-methoxy-, bluish red; 4-hydroxy-3-methoxy-, violet-blue; 3-hydroxy-4-acetoxybenzene, cherry-red; BzH and "nitrobenzaldehyde," yellowish. No color is produced by PhOH and the 3 dihydroxybenzenes. 3,4-Dihydroxybenzaldehyde, under the above conditions, gives with the following alcs. the colors mentioned. They refer to transmitted light, the colors in reflected light are given in parentheses (): MeOH, deep scarlet (steelblue); EtOH, deep scarlet (bluish violet); PraOH, deep scarlet (lilac); amyl alc., deep scarlet (bluish lilac); benzyl alc., red (olive-green); glycerol, reddish (olive-green); Pr<sup>\$</sup>OH, deep pine-green; sec. isoamyl alc., dark green; tert. butyl alc., colorless. The cause of this production of color is not understood and, in the case of complicated alcs., the colors (if any) cannot be used to differentiate between primary, secondary and tertiary alcs. p-Hydroxybenzaldimethylacetal, HOC,H,CH(OMe), is prepared from p-hydroxybenzaldehyde, Me orthoformate, highly purified MeOH and dry HCl; the mixture is b. during 1 min., cooled quickly and the acid neutralized exactly; crystals from Et<sub>2</sub>O + ligroin, m. 60-5°. It is rather unstable. Yield, 87%. m-Hydroxybenzaldimethylacetal is prepared in a similar manner to the preceding compd.; liquid, b, 150°, mostly decomp. o-Hydroxybenzaldimethylacetal, prepared like the isomers; oily, based about 65° (some decomp.). Acetals could not be obtained from 3,4-dihydroxybenzaldehyde, or from its carbonate, nor from 2,4-dihydroxybenzaldehyde. The 2,5- and 2,3-dihydroxybenzaldehydes are, in part, converted into resins, but they likewise fail to form acetals. The o- and p-hydroxybenzaldimethylacetals eliminate MeOH, when heated, the residues are resinous. The following mercaptals, HORC<sub>6</sub>H<sub>2</sub>CH(SMe)<sub>2</sub> (R = OH or H), were prepared from the resp. aldehydes, Et<sub>2</sub>O, dry HCl and Zn methyl- or p-nitrobenzylmercaptans, in a freezing mixture. m-Hydroxybenzaldimethylmercaptal, oil. Yield, 116% of the aldehyde. p-Hydroxybenzal dimethylmercaptal, thick plates with acute angles from Et<sub>2</sub>O + petroleum ether, m. 73.5°. Yield, 130% of the aldehyde. 3,4-Dihydroxybenzaldimethylmercaptal, colorless, lustrous plates from H<sub>2</sub>O, m. 108-9°. Yield, 102% of the aldehyde. This mercaptal is also formed when the cyclocarbonate mercaptal is hydrolyzed by means of aq. pyridine. 2,3-Dihydroxybenzaldi-4'-nitrobenzylmercaptal, (HO)<sub>2</sub>C<sub>6</sub>H<sub>2</sub>CH(SCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>NO<sub>2</sub>)<sub>2</sub>, yellow prisms from glacial AcOH, m. 177°. Yield, about 157% of the aldehyde. 2,3-Dihydroxybenzaldimethylmercaptal, liquid. 2,5-Dihydroxybenzaldimethylmercaptal, viscid oil. 2,5-Dihydroxybenzaldi-4'-nitrobenzalmercaptal, (HO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CH(SCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>NO<sub>2</sub>)<sub>2</sub>, large, green prisms from glacial AcOH, m. 170°. Yield, 300% of the aldehyde. The above mercaptals all decomp. when heated, even under greatly reduced pressure; S compounds are evolved, of which at least 75% consists of methylmercaptan. The non volatil residue is resinous. No mercaptal could be obtained from 2,4-dihydroxybenzaldehyde; it gave only tarry matter. The naphthocinchonic acids were prepared by warming the resp. aldehydes with  $\beta$ -naphthylamine, pyruvic acid and alc. The yield from salicylic aldehyde and from 4-hydroxy-

3-methoxybenzaldehyde is 89 and 55%, resp. 3-Hydroxyphenyl-β-naphthocinchonic acid, C<sub>20</sub>H<sub>12</sub>O<sub>2</sub>N, from m-hydroxybenzaldehyde; yellow, crystallin powder m. 284.5°, previously softening. Yield, 78%. 4-Hydroxyphenyl-β-naphthocinchonic acid, from p-hydroxybenzaldehyde; light yellow, crystallin powder, m. 325.5°. Yield, 40%. 3-Hydroxy-4-methoxyphenyl-β-naphthocinchonic acid, CnH16O4N, from 3-hydroxy-4methoxybenzaldehyde; lemon-yellow, crystallin powder, m. 295°. Yield, 80%. 2-Hydroxy-3-methoxyphenyl-β-naphthocinchonic acid, from 2-hydroxy-3-methoxybenzaldehyde; almost white, crystallin powder, m. 251°. Yield, 93%. 3,4-Dihydroxyphenyl-β-naphthocinchonic acid, C20H12O4N, from 3,4-dihydroxybenzaldehyde; orange-yellow, crystallin powder, m. 317.5°. It gives a brownish, wine-red color with FeCl, in presence of Na<sub>2</sub>CO<sub>2</sub>. Yield, 55%. No condensation took place in the case of 2,3-2,4- and 2,5-dihydroxybenzaldehydes. 2-Propenylphenyl, HOC,H,CH: CHMe, is formed from salicylic aldehyde and EtMgI, in Et,O; silky needles from ligroin, m. 34.8°; solidifies 34.3°; b<sub>788</sub> 229-31°. It attacks the skin, has the odor and taste of PhOH, gives a colorless soln. with aq. alkali hydroxides and exhibits the propenyl reaction with Hg(OAc)<sub>2</sub>. The propenylphenol decolorizes at least 4 ats. of Br, finally evolving HBr, and forming a pseudobromide; small crystals from ligroin. It is insol. in alkalies. A compound, possibly (CoH10O), of high b. p. is formed together with the propenylphenol; viscid, pale yellow oil. Like the propenylphenol, it gives a yellowish red color with conc. H<sub>2</sub>SO<sub>4</sub>, is sol. in aq. NaOH and decolorizes Br, in CHCl<sub>2</sub>, HBr being evolved. Yield, about 50% of the aldehyde. About 92% of the aldehyde interacts with the EtMgI. 2-Propenyl-6-methoxyphenol, HOC, H, (OMe) CH: CHMe, is prepared from 2-hydroxy-3-methoxybenzaldehyde and EtMgI, in the manner described above and is purified from I compounds by adding anhydrous pyridine to the crude phenol, in Et<sub>2</sub>O; silky lustrous needles from ligroin, m. 81°; b<sub>12</sub> 137-40°. It resembles the preceding compd. in its general properties and reactions. In conc. H<sub>2</sub>SO<sub>4</sub> the color is blood-red; with FeCl<sub>2</sub>, fugitive blue. The phenol is moderately volatil with steam. Yield, 50% of the aldehyde; a viscid compound is also formed together with the preceding substance, so that the total quantity of aldehyde which reacts is about 85%. Sodium salt, white plates. Bromo-2-propenyl-6-methoxyphenol dibromide, C10H11O2Br2, from the phenol and Br, in Et2O; plates from petroleum ether, m. III°. 2,3-Dimethoxyphenylmethylcarbinol, (MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CHMeOH, from 2,3-dimethoxybenzaldehyde and MeMgI; colorless liquid, b<sub>14</sub> 151-2°; d<sup>20</sup> 1.1213. Yield, 93%. Phenylurethan, colorless needles from C<sub>6</sub>H<sub>6</sub>, m. 127°. Yield, 100% of the carbinol. Tetramol.-2,3-dihydroxypropenylbenzene, (C<sub>2</sub>H<sub>10</sub>O<sub>2</sub>)4, is obtained from 2,3dihydroxybenzaldehyde and EtMgI and is purified by means of anhydrous pyridine, the resulting oil eliminates H<sub>2</sub>O at 190° (12 mm.); brown, amorphous powder from C<sub>a</sub>H<sub>a</sub>; from other solvents it is deposited as a varnish, softens about 95°; mol. wt., in b. Et<sub>2</sub>O, 615. Yield, about 85%. It gives the pyrocatechol reactions and in conc. H<sub>2</sub>SO<sub>4</sub> forms a fuchsine-red soln. At a high temp, pyrocatechol sublimes. The expts. described below were made by dissolving the resp. aldehydes (0.1 g.) in H<sub>2</sub>O (3 cc.) and adding fuchsine-SO<sub>2</sub> soln. (5 cc.). In the case of the dihydroxy aldehydes CHCl<sub>2</sub> (1 cc.) was also added, because, after the mixture has been shaken, the coloring matter dissolves in the CHCla, so that the delicacy of the test is increased. The fuchsine soln. was prepared according to the directions of Woodman and Lyford (C. A., 2, 3383). The numbers given below in roman type express the time in seconds required for the production of a color; those in parentheses the time in seconds required to attain the maximum color intensity; in italics are given the number of min. necessary for the discharge of the color. BzH, 1 (30) 30 (feeble); o-hydroxybenzaldchyde, 5 (30) 10; m-hydroxybenzaldehyde, 5 (30) 10; p-hydroxybenzaldehyde, after 2 min. hardly recognizable color which disappears rapidly; p-methoxybenzaldehyde, 12 (60) 15; 2,3- and 2,4-dihydroxybenzaldehydes, no distinct color; 2,5-dihydroxybenzaldehyde 10 (—) 3; 3,4-dihydroxybenzaldehyde, no color; 3-hydroxy-4-methoxybenzaldehyde, 60 (—) 15 (feeble); 4-hydroxy-3-methoxybenzaldehyde, 45 (—) 15 (feeble); 3,4-methyleneoxybenzaldehyde, 15 (20) 15 (feeble) (see following abstr.).

J. B. T.

Nature of Phenolic Aldehydes. II. Reactivity of the Phenolic Groups in the Phenolic Aldehydes. H. PAULY, KONRAD SCHÜBEL AND KARL LOCKEMANN. Chem. Inst., Univ. Würzburg. Ann., 383, 288-337; see preceding abstr.—The cond. detns., the results of which are given below, were made at 25°, in H<sub>2</sub>O; only the mean values for K are reproduced. In the expts. with pyrocatechol the electrodes were b. with H<sub>2</sub>O between each detn. in order to prevent oxidation. Pyrocatechol,  $\mu = 381$ ; K 3.5  $\times$ 10<sup>-10</sup>. p-Hydroxybenzaldehyde, n ∞ 380; K 2.2 × 10<sup>-8</sup>. m-Hydroxybenzaldehyde,  $\mu = 380$ ; K 1.0 × 10<sup>-8</sup>. 3,4-Dihydroxybenzaldehyde,  $\mu = 379$ ; K 2.8 × 10<sup>-8</sup>. Detns. have been made of the relative velocity of hydrolysis of pyrocatechol and of 3.4-dihydroxybenzaldehyde cyclocarbonates, by means of aq. pyridine, at 50°. The velocities are in the ratio pyrocatechol: carbonate::1:75. The values 1.0  $\times$  10<sup>-7</sup>,  $1.8 \times 10^{-8}$  and  $8 \times 10^{-8}$  were obtained for the dissociation constants of o-, m- and p-hydroxybenzaldehydes, resp., by titration in MeOH, with aq. NaOH, in presence of phenolph. In 20% alc., with 0.1 N aq. NaOH and phenolph. 2,3-, 2,4-, 2,5- and 3,4-dihydroxybenzaldehydes are all monobasic. 2,3-Dihydroxybenzaldehyde is best prepared by gently b. 2-hydroxy-3-methoxybenzaldehyde with a slight excess of HBr, in glacial AcOH, the progress of the reaction being followed by collecting the MeBr over brine; the residue is neutralized by means of yellow PbO, in glacial AcOH, and the liquid, after filtration, is conc. and distilled under reduced pressure, The aldehyde b<sub>16</sub> 119-20°; m. 235° (754 mm.). Yield, of pure compd., 45% of the MeO derivative. Lead and aluminium salts, yellow. Copper salt, ochre-brown. Cobalt salt, rose-brown. All were prepared from the Na salt. The aldehyde is best recrystallized from ligroin; from C<sub>a</sub>H<sub>a</sub> unstable crystals containing solvate are deposited. The aldehyde combines with "phenyldiazoniumsulfonic acid," in presence of Na<sub>2</sub>CO<sub>2</sub>. Anil, (HO)<sub>2</sub>-C.H.CH: NPh, scarlet needles with a blue reflex from alc., m. 135°. In alkalies the color is yellowish orange and in HCl, canary-yellow. β-Naphthylamine derivative, (HO), C.H.CH: NC10H2, slender bluish needles from alc., m. 164°. Benzidine derivative, (OH), C.H.CH: NC,H.C.H.N: CHC,H. (OH), carmin-colored, insol. powder, does not m. Phenylhydrazone, C12H12O2N2, pale yellow needles from alc. (50%), m. 167°, not 176°. Semicarbazone, C.H.O.N., white plates, m. and darkens 226° (cor.) (decomp.). Sodium hydrogen sulfite derivative, white plates. In presence of pure, glacial AcOH, the aldehyde and malonic acid form a compound; slender, pale yellow needles from H<sub>2</sub>O, m. about 200°. The aldehyde also condenses with indanedione. The following derivatives of 2,3-DIHYDROXYBENZALDEHYDE have been prepared. Barium salt, C,H,O,Ba, prepared in alc., from Ba(OH),; the ppt. is at first brick-red, but quickly changes to orange. After drying in air it contains 4 H<sub>2</sub>O; at 105°, 2 H<sub>2</sub>O and it becomes anhydrous at 170-80°. 2-Carbomethoxyoxy-3-methoxybenzaldehyde, MeO<sub>2</sub>COC<sub>2</sub>H<sub>2</sub>(OMe)CHO, is prepared by treating 2-hydroxy-3-methoxybenzaldehyde with KOH, in dil. alc. and warming the resulting K salt with C.H. and Me chlorocarbonate; colorless, hexagonal plates from ligroin + C<sub>8</sub>H<sub>6</sub> (9: 1), m. 60°; b<sub>12</sub> 170-6°. 2,3-Dimethoxybenzaldehyde, b<sub>74</sub> 256°; b<sub>12</sub> 137°. Benzaldehyde 2,3-

cyclocarbonate, OC C<sub>2</sub>H<sub>2</sub>CHO, from the Ba salt and COCl<sub>2</sub>, in toluene; colorless

needles m. 105°; b<sub>16</sub> 164°. Yield, 25% of the ester. With FeCl<sub>3</sub>, in alc., it gives a brownish yellow color. 3-Carbomethoxyoxy-2-hydroxybenzaldehyde, HOC<sub>2</sub>H<sub>3</sub>(CHO)-OCO<sub>2</sub>Me, is formed by b. the preceding compd. with MeOH, or from Me chlorocarbonate and Na 2,3-dihydroxybenzaldehyde. With Na<sub>2</sub>CO<sub>3</sub> and "phenyldiazoniumsul-

fonic acid" it gives an orange color, changing to lemon-yellow on acidification. A saturated, ag. soln. of KHCO, is well adapted for use in the methylation of phenol derivs. which are sensitive to O. 3-Carbomethoxyoxy-2-hydroxybenzaldehyde could not be methylated. Copper sall, Cu(C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>), from Cu(OAc), in MeOH; olive-yellow plates. Anil, HOC, H, (OCO, Me)CH: NPh, egg-yellow crystals from MeOH, m. 72°. It readily forms 'colloidal solns. The following derivatives of 3,4-DIHYDROXYBENZ-ALDEHYDE have been prepared. Barium salt, C7H4O2Ba, is prepared like its isomer, orange colored, after drying at 160-70° during several hrs. After drying during 10 mins. at 160° it contains 1H<sub>2</sub>O and at 120° it consists of egg-yellow scales with 2 H<sub>2</sub>O. Acid salt, Ba(C, H<sub>2</sub>O<sub>2</sub>), pale greenish yellow needles with 3 H<sub>2</sub>O. It is prepared in a similar manner to the neutral salt by the use of 0.5 equivalent of Ba(OH). With MerSO4 the Ba salt gives 3-methoxy-4-hydroxy- and 4-methoxy-3-hydroxybenzaldehydes, in equal quantity; total yield, 55% of the Ba salt. With alc. KOH and MeI, the yield of the 2 mono ethers is in the ratio 4-MeO: 3-MeO:: 9:1. 3-Methoxy-4carbomethoxyoxybenzaldehyde, MeOCaHa(CHO)OCOaMe, is prepared from 3-methoxy-4hydroxybenzaldehyde, KOH and Me chlorocarbonate; colorless crystals from MeOH (50%), m. 91.5°. Yield, 66%. 4-Methoxy-3-carbomethoxyoxybenzaldehyde could not be prepared in the manner just described, but it is formed without difficulty by treatment of the Na salt, from Et<sub>2</sub>O, MeONa and 4-methoxy-3-hydroxybenzaldehyde, with C.H. and Me chlorocarbonate; large, silky lustrous crystals from H.O, m. 121°. It is not sol. in cold, aq. alkali hydroxides. Benzaldehyde 3,4-cyclocarbonate and PhN, Ha, in glacial AcOH, at the ordinary temp., give the phenylhydrazone, CO2: C2H2CH: NNHPh; light yellow crystals from glacial AcOH, m. 177°. When the preceding constituents are warmed together the product consists of the phenylhydrazonephenylhydraside, PhNHNHCO2C6H2(OH)CH: NNHPh; white plates from glacial AcOH, m. 186°. Yield, 200% of the carbonate. Semicarbazone, CO.: C.H.CH: NNH-CONH., prepared in glacial AcOH; white plates from glacial AcOH, m. 220° (decomp.). Methylmercapial, CO<sub>2</sub>: C<sub>2</sub>H<sub>2</sub>CH(SMe)<sub>2</sub>, from the carbonate, methylmercaptan and dry HCl; large plates of the monoclinic system from C<sub>8</sub>H<sub>6</sub> + ligroin (1:1), m. 56.5°; b<sub>18</sub> 223°. Yield, quant. With hydrolyzing agents it evolves CO<sub>2</sub> and forms 3,4-dihydroxybenzalmethylmercaptal. In presence of HCl, the cyclic carbonate condenses with acetone, in glacial AcOH; the resulting compound is deposited in steel-blue crystals which, in contact with porous earthenware, become dark red, when washed with acetone the color changes to chrome-green and on warming it turns light yellow. In glacial AcOH + HCl the yellow crystals give an intense fuchsine-red color. Diacetate, CO<sub>2</sub>: C<sub>2</sub>H<sub>2</sub>CH(OAc)<sub>2</sub>, from the carbonate, Ac<sub>2</sub>O and a little conc. H<sub>2</sub>SO<sub>4</sub>; it is identical with the compd. from AgOAc and "dichloropiperonaldehyde." 3-Carbomethoxyoxy-4-hydroxybenzaldehyde, HOC<sub>2</sub>H<sub>2</sub>(OCO<sub>2</sub>Me)CHO, is prepared by b. the cyclic carbonate with MeOH, during 3 hrs. and is identical with the compd. from "dichloropiperonalaldehyde," MeOH and AcONa (C. A., 3, 2555); m. 96° not 93°. With Me,SO, and NaHCO, it gives the 3-carbomethoxyoxy-4-methoxybenzaldehyde described above. 3-Carbomethoxyoxy-4-hydroxybenzaldehyde is not identical with "methyl pyrocatechnicaldehydecarbonate," m. 99° (Ger. Pat. 93178); when distilled the 3-carbomethoxyoxy compd. regenerates the original cyclocarbonate (m. 123°). 3-Carbopiperidido-4-hydroxybenzaldehyde, CaH10NCO2CaH2(OH)CHO, is formed from the cyclocarbonate and piperidine, the product being subsequently acidified; needles from alc., m. 123°. With conc. HCl it evolves CO<sub>2</sub>. Phenylhydrazone, C<sub>19</sub>H<sub>21</sub>O<sub>3</sub>N<sub>2</sub>, from the preceding compd and PhN, H, in glacial AcOH; plates from glacial AcOH, m. 159°. The nature of the above piperidide is further shown by the fact that, when treated with Me<sub>2</sub>SO<sub>4</sub> and "bicarbonate" and the product hydrolyzed by means of H<sub>2</sub>SO<sub>4</sub> (10%) 3-hydroxy-4-methoxybenzaldehyde is formed. Salicylaldipiperyl, HOC.H.CH(NC.H.a) is obtained from salicylic aldehyde and piperidine; large, colorless plates from acetone, m. 87-8°. It speedily becomes red on exposure to air and is hydrolyzed easily by alkalies. 2,5-Dihydroxybensalsemicarbasone, (HO)<sub>2</sub>C<sub>6</sub>H<sub>6</sub>CH: NNHCONH<sub>3</sub>, is prepared in aq. AcONa; slender crystals, m. 249°. A yellow color is produced when salicylic aldehyde, MeI, pure MeOH and Ag<sub>6</sub>O (free from alkali) are mixed. The only substance which could be isolated from the reaction products was methoxysalicylic aldehyde. Yield, about 15%.

J. BISHOP TINGLE.

Fusibility Curves of Gaseous Mixtures (BAUME, PERROT). 2.

Reduction in Presence of Finely Divided Palladium (BRETEAU). . 6.

Reactions for Formation of Iodine Derivatives (GEHARD). 17.

Rhizome of Aschlepias vincetoxicum (MASSON). 17.

Action of Various Acids on Starch and Dextrin (CONINCE, RAYNAUD). 28.

Compounds of Basic Dyestuffs with Tannin (SANIN). 25.

ANGER, E.: Ueber die Gewinnung des o-Menthons-5 und Studien zur Bildung von o-Menthon-3 und o-Menthon-6. Hildesheim: A. Lax. 86 pp., 2 M.

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ELLINGER, P.: Untersuchungen in einfach ungesättingten Kohlenwasserstoffen, Sauren und Estern mit semicyclischer Doppelbindung. Heidelberg: I. Hörning. 8°, 1.20 M.

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## 11. BIOLOGICAL CHEMISTRY.

wm. j. gies. GENERAL.

Enzymic Hydrogen Peroxide Decomposition. I. PERCY WAENTIG AND OTTO STECHE. Lab. Angew. Chemie, Univ. Leipzig. Z. physiol. Chem., 72, 226-304.— The decomp. of H<sub>2</sub>O<sub>2</sub> by blood enzymes is not a reaction of the first order. The purity of the enzyme has but slight influence on the rate of reaction. Enzymes freed of inactive substance are more susceptible to outside destructive influences. The optimum at 0° is with neutral reaction. The CO<sub>2</sub> of ordinary dist. H<sub>2</sub>O or slight amts. of alkali sufficient to turn phenolph. reduces very decidedly the rate of reaction. At higher temp. slight acidity favors the rate.

G. M. Meyer.

The Pseudo-peroxidase Reaction between Hemoglobin, its Derivatives and Guaia-conic Acid (Guaiacum Reaction for Blood Pigment). G. A. BUCKMASTER. 7th Intern. Congr. Applied Chem., 1909 (Sect. IV, A, 2), 30; J. Chem. Soc., 100, I, 390.

J. J. M.

Experiments on the Diffusibility of Cholesterol Esters and of Lecithin Compounds. E. Boas and J. Rosenbloom. Lab. Biol. Chem., Columbia Univ. Proc. Soc. Exp

Biol. Med., 8, 132-3.—Cholesterol benzoate, cholesterol stearate, cholesterol oleate, and cholesterol palmitate, dissolved in Et<sub>2</sub>O, readily diffuse through rubber into Et<sub>2</sub>O. Cholesterol stearate with a mol. wt. of 652.61 diffuses, whereas the various lecithins, with mol. wts. of 770-785 do not. If it is assumed that the diffusion of a substance depends on the size of its mols., the above fact would strengthen Hiestand's figure of 1446 obtained by a mol. wt. detn. for egg-yolk lecithin. Compds. of lecithin with glucose, lactic acid, strychnine, digitonin, salicin, urea, creatine, creatinine and caffeine were prepared. The glucose and lactic acid dialyzed completely, the strychnine, digitonin and salicin dialyzed partially, while urea, creatine, creatinine and caffeine did not dialyze at all.

V. C. Myers.

Precipitation of Proteins. A. J. J. VANDEVELDE. Bull. soc. chim. belg., 25, 166-73; cf. C. A., 2, 1309; 5, 901.—The results obtained from pptg. and sepg. the proteins of milk, of colostrum, of serum of beef and of the horse, and of white of egg are tabulated.

I. K. Phel. Ps.

The Permeability of Living Cells to Salts in Pure and Balanced Solutions. W. J. V. OSTERROUT. Science, 34, 187-9.—The author does not agree with Overton (in expts. on Spirogyra) that only those substances penetrate the cell which are sol. in lipoids. The common method of detg. osmotic pressure by plasmolyzing in Na and K salts is not trustworthy. Ca salts give more nearly the true osmotic pressure. Since one substance may greatly affect penetration by another it is unsafe to add a toxic to a non-toxic substance and to judge the penetration by the former from the plasmolytic action of the mixt. The membrane of the plasma is not of lipoid but of protein nature. By mixing solns. of NaCl and CaCl<sub>2</sub>, which taken separately are not strong enough to plasmolyze a soln., decided plasmolytic properties are obtained. G. M. MEYER.

Knowledge of Lecithin (COHN). 12.

Electrochemistry of Proteins (ROBERTSON). 2.

Action of Salts in Heterogeneous Systems (SCHRYVER). 2.

### METHODS AND APPARATUS.

Physical-chemical Investigations of Animal Liquids. IV. Technic of the Electrometric Method for the Study of the Reactions of Liquids of the Organism. G. QUAGLIARIELLO. Ist. fisiol. sper. r. univ. Napoli. Atti accad. Lincei, 20, 11, 107-11.—The results of e. m. f. measurements on blood, using Bugarzsky's (Z. anorg. Chem., 16) and Bjerrum's methods (Z. physik. Chem., 53, 428), resp., to eliminate the diffusion potential, are always slightly higher (about 0.0025 v. or 0.8%) by the first method; the author is of the opinion that the second gives the more nearly correct values.

CHAS. A. ROUILLER.

The Reagents Employed for the Determination of Blood Stains in Legal Medicin. Bordas. Compt. rend., 153, 302-3.—The benzidine reaction (Rudolf Adler reagent) for blood stains, like numerous others (Meyer's reagent, see Sartory, C. A., 5, 3465) is of value only in so far as a negative reaction would save further unnecessary exam. A positive test with this reagent can not be considered conclusive. G. M. MEYER.

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BUCHNER, G.: Merkblatt für Harn- und andere physiologisch- und pathologischchemische, mikroskopische und bakertioskopische Untersuchungen. München: H. Lukaschik. 8°, 12 pp., 50 Pf.

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# BACTERIOLOGY.

ABEL, R.: Bakteriologisches Taschenbuch. 15 Aufl. Wurzburg: A. Stuber. 8°, 137 pp., 2° M.

#### BOTANY.

An Hydrometric Investigation of the Influence of Sea Water on the Distribution of Salt Marsh and Estuarine Plants. J. W. HARSHBERGER. Proc. Am. Phil. Soc., 50, 457-96.

Aldehyde in Leaves. Hartwig Franzen. Heidelberg. Chem. Ztg., 34, 1003-4. —By distilling 600 kg. horn-beam leaves 200 g. of distillate were obtained. By ultimate analyses of the condensation products with m-nitrobenzoylhydrazine and benzoylhydrazine of the free aldehyde present and of the Ag salt of the acid obtained by oxidation of the aldehyde with Ag<sub>2</sub>O, the formula of the aldehyde was calc. to C<sub>2</sub>H<sub>2</sub>CHO. Ag<sub>2</sub>O also gave a solid acid which is identical with Fittig and Baker's  $\alpha,\beta$ -hexylenic acid, CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH : CHCOOH, prepared from hydrosorbic acid. The dibromide was identical with  $\alpha,\beta$ -dibromocaproic acid. Therefore the aldehyde must be  $\alpha,\beta$ -hexylenic aldehyde. Leaves of all plants examined contain the aldehyde; chestnut, grape, brake, maple, oak, lupin, clover, common beech, hazel, walnut and raspberry contained it in large quantity. It seems safe to assume its presence in all plants. The aldehyde differs from the hexose aldoses in lacking the 5 OH groups. Its significance in plants and its relation to sugar synthesis are under investigation.

E. J. WITZEMANN.

#### PHYSIOLOGY.

Organic Matter in the Expired Breath. MILTON J. ROSENAU AND HAROLD L. Amoss. Dept. of Preventive Med. and Hyg., Harvard. J. Med. Res., 25, 35-85.—The authors demonstrated the presence of organic matter (protein in nature) in the condensed moisture from the expired breath of man by means of the anaphylaxis reaction in guinea pigs. Of 99 pigs, using human blood serum for the tests, 26 gave definit symptoms of anaphylaxis, death occurring in 4. The expired breath of 8 people was tested and 5 of these gave positive reactions.

H. J. CORPER.

Physiology of the Blood Plates. J. Loeber. Med. Poliklinik, Freiburg i/B. Arch. ges. Physiol., 140, 281-92.—Blood, rendered non-coagulable by hirudin, shows regularly a consumption of O 2-3 times as great as defibrinated blood. This phenomenon is not attributable to the hirudin nor to the metabolism of the leucocytes. It is due to the respiratory gas metabolism of the blood plates. This observation is incompatible with the view still expressed by many, that these form elements which arize from the circulating erythrocytes. On the other hand it does not speak for the independent cell nature of the plates.

Walter A. Jacobs.

The Formation of Bile Pigments from Blood. Theodor Brugsch and Yoshimoto. Z. exp. Path., 8, 639-44; through Chem. Zentr., 1911, I, 994.—By the quant. estn. of bile pigment excreted in the urin, feces and gall of animals with artificial gall-fistula and ligated ductus choledochus, the author studied the genetic relationship between bile pigment and hematin. Before and after the hemin injection (0.4% alk. soln. of hemin-HCl daily) a mean biliverdin excretion of 0.37 g. per day was observed; during the hemin period this increased about 0.04 g. 2 mg. of hemin passed out in the urin during the hemin period, as against 1.6 and 1.7 mg. for the pre- and post-hemin periods. These facts indicate the formation of bile pigment from hematin according to the equation:  $C_{22}H_{22}N_4Fe + 2H_2O = C_{22}H_{23}N_4O_6 + Fe$ . For the urobilin detn. in the urin an acid ether-alc. soln. of urobilin was prepared according to F. Muller and Huppert and spectrophotometrically examined in a König-Martens app. For the detn. in the feces the 24 hr. feces were extracted with alc. the alc. soln. conc. to 10 cc. dild. with 50-

100 cc. water, filtered, shaken out with 3-4 vol. petroleum ether and then detd. in acid ether-alc. soln. For detg. bilirubin in the gall, the bilirubin was converted by oxidation in the air to biliverdin and this extracted with alc. acidified with H<sub>2</sub>SO<sub>4</sub>. After conc. to a syrup the ext. was dissolved in conc. H<sub>2</sub>SO<sub>4</sub> and water and pptd. with H<sub>2</sub>O as a green flocculent ppt.; was filtered, washed and extracted with abs. alc. On evapn. the biliverdin remained as a dark green amorphous substance and was colorimetrically detd. in 0.05% acid alc. soln. Influence of Hematoporphyrin, Hemin and Urobilin on the Formation of Pile Pigment. Theodore Brugsch and K. Kawashima. Z. exp. Path., 8, 645-8; Chem. Zentr., 1911, I, 995.—The influence of hematoporphyrin on the excretion of biliverdin was studied in a dog with a gall fistula, and then further the influence of hemin and urobilin. Hematoporphyrin is surely in part not converted into bile pigment but is excreted as such through the gall. It is undecided whether it is converted into urobilin. Hemin increases the production of bile pigment (likewise urobilin), being oxidized to bilirubin or biliverdin.

Walter A. Jacobs.

Presence of Nuclease and Connectivase in the Gastric Contents of Infants. Mariano Mittra. Palermo. Folia clinica, 3, 274-8.—An infant a year old was fed on milk, on milk with flour, and on porridge made from legumes and at intervals of 0.5, 1, 1.5 and 2 hrs. after ingestion portions were removed and tested with connective tissue and muscle fiber. No nuclease nor connectivase was found. A similar expt. on a child of 15 mos. seemed to show the presence of both.

I. K. Phelps.

Ereptase of the Intestinal Juice. GIUSEPPE AMANTEA. Lab. fisiol. r. univ. Roma. Atti accad. Lincei, 20, II, 74-8.—Dogs were subjected to the Vella intestinal ansa operation and the intestinal juice, stimulated by mechanical means (glass pellets, etc.) or chem. reagents (CHCl<sub>2</sub>, HCl, AcOH, HCl or oleic acid in bile, pilocarpine, peptone) in direct contact with the enteric mucous membrane, was tested with peptone and casein solns., Sörensen's method (C. A., 2, 1288) being used to det. the amino acids formed. The author concludes that even isolated intestinal segments (therefore not in contact with the pancreatic juice) contain an ereptase. The ereptic activity of juice stimulated mechanically or by oleic acid in bile is greatest, that stimulated by 0.5% HCl least, but the differences are not marked, and even the same stimulant may show differences. In general, more amino acids are formed from peptone than from casein.

Chas. A. Roulller.

Study of the Osmotic Work of the Kidneys. X. The Product of the Glomeruli is a Blood Filtrate. ERNST FREY. Pharm. Inst., Univ. Jena. Arch. ges. Physiol., 139, 435-64.-- A few expts. without the ingestion of NaBr yielded results formerly indicated (C. A., 5, 103). During caffeine diuresis the urin approaches in its comp. that of the serum as shown by detns, of its total conc. ( $\Delta$ ) and its Cl content. This occurs whether the urin of the animal is poor or rich in chlorides. In salt (NaNO<sub>2</sub>) diuresis, likewise, without regard to the salt or H<sub>2</sub>O content of the body, with increasing diuresis the chloride content of both urin and blood and the depression of the f. p. approach one another. Whether the animal is rich or poor in H<sub>2</sub>O the injection of dil. or conc. NaCl solns. resp. is followed by the same result. The product of the glomeruli is therefore a filtrate of the blood. The point of attack of these diuretics is in the glomeruli, these vessels being widened and the filtration of the blood through them increased. XI. The Resorption of Water in the Uriniferous. Tubules Corresponding to the Total Concentration. Ibid., 465-511.—The facts above shown are here proved to be quant. If the conc. of the urin is double that of the serum, then the urin contains twice as much NaCl as the serum. Where the animal is rich in NaCl there is no reabsorption of NaCl nor is any added to the filtrate of the glomeruli by secretion from the tubules. The path of NaCl is alone one of filtration. XII. Iodide, Nitrate, Sulfate and Phosphate Reach the Uriniferous Tubules by Secretion. Ibid., 512-31. NaCl and NaBr

are the only substances which reach the urin from the blood by filtration; these salts are also reabsorbed under ordinary conditions. The removal of all other salts occurs by secretory processes in the tubules. With NaNO<sub>3</sub>, filtration besides secretion occurs. These substances are not reabsorbed. If the urin salts for which a filtration would first be considered, are removed by secretion, then the removal of the sp. urin constituents such as urea and uric acid is most likely also a secretory process. XIII. The Retention of Sodium Chloride, a Phenomenon of Interchange between Filtered and Secreted Substances. *Ibid.*, 532-47. The retention of NaCl appears as an antagonism between filtered and secreted substances. If NaCl retention is observed in pathology, it is not a sign of a primary inability of the kidneys to excrete NaCl, but evidence that substances which seek the urin are accumulating in the blood.

WALTER A. JACOBS.

HÖBER, R.: Physikalische Chemie der Zelle und der Gewebe. 3 Aufl. Leipzig: W. Engelmann. 19.25 M.

#### PATHOLOGY.

Inhibition of Transudate and Exudate Formation by Calcium Salts. RICHARD CHIARI AND HANS JANUSCHEE. Pharm. Inst., Univ. Vienna. Arch. exp. Path. Pharm., 65, 420–7.—Pleural exudate produced by poisoning with NaI, thiosinamine and diphtheria toxin (in dogs and guinea pigs) and also inflammatory edema of the conjunctiva (rabbit) following administration of mustard oil and abrin could be inhibited or greatly reduced by the copious administration of Ca salts. The most effective is CaCl<sub>2</sub> and then the lactate. The inhibition of exudate occurs 3 hrs. after intravenous injection; in 24 hrs. after subcutaneous injection it disappears. This action is independent of the tendency of Ca salt to induce coagulation.

Walter A. Jacobs.

The Influence of Calcium Salts upon Exudate Formation. RICHARD LEVY. Chirurg. Universitätsklinik in Breslau. Berl. klin. Wockschr., 1911, 1322-4.—The author could find no constant inhibitory effect of Ca salts upon the production of exudates and transudates in guinea pigs by means of toxic doses of diphtheria toxin, and thus could not corroborate the observations of Chiari and Januschke. CaCl. had no influence upon the production of hemorrhages in mice by means of Crotolus poison.

H. J. CORPER.

Salt Fever. HERMAN FREUND. Med. Klinik., Univ. Heidelberg. Arch. exp. Path. Pharm., 65, 225-38.—Salt fever is greatly dependent upon the disposition of the animal. Comparison of the action of adrenaline and NaCl showed striking analogy. From this a common point of attack for the two may be assumed. The disposition towards salt fever may be defined as an increased excitability or tonus of the sympathicus.

Walter A. Jacobs.

The Effect of Quinine and Quinine Derivatives on Experimental Trypanosome Infection. J. Morgenroth and L. Halberstaedter. Pathol. Inst., Berlin. Sitzb. kgl. preuss. Akad. Wiss., 1911, 30-7.—In a previous paper (Ibid., 1910), the authors have shown that quinine and its derivs. exert a preventative action towards infection with Trypansoma bruces. In the trials here reported a comparative study of quinine and hydroquinine as antitrypanosome drugs was made by feeding the compds. to mice which were subsequently injected with trypanosomes. Hydroquinine was much the more powerful of the two to prevent the infection and was the less toxic to the animal. In hydroquinine the double bond of the side chain does not exist, thus: CH — CH = CH<sub>2</sub> (quinine) and CH — CH<sub>2</sub> — CH<sub>3</sub> (hydroquinine).

A Possible Significance of the Cammidge Reaction. L. B. STOOKEY. Physiol. Lab., Med. School, Univ. Southern California. Proc. Soc. Exp. Biol. Med., 8, 138.—From expts. made thus far the author considers it probable that in cases showing a

positive Cammidge reaction there may be some relationship between the amt. of cane sugar ingested and the intensity of the reaction.

V. C. MYERS.

Hemoglobin Metabolism and the Clinical Significance of Extraction of Bilinogen.

L. Zoja. Med. Klinik, Parma. Folia Hematol., 12, 1-22.—Polemic against Brugsch.

H. C. W.

The Clinical Significance of Excretion of Urobilinogen. Th. BRUGSCH. II Med. Klinik, Univ. Berlin. Folia Hematol., 12, 23-5.—Reply to the above. H. G. W.

Histological and Functional Behavior of the Suprarenal Glands in Starving Rabbits. Franz Lucksch. Pharm. Inst., Univ. Prague. Arch. exp. Path. Pharm., 65, 161-3.

—In preps. from a series of starving rabbits the medullary substance of the supra-renal glands were chromated normally. Chromaffinous tissue is not essentially affected by starvation, as is the case in various forms of intoxication. Walter A. Jacobs.

ERLENMEYER, E.: Blei- und Eisenbilanz bei experimenteller, chronischer Bleivergiftung. Bonn: Carl Georgi. 1.50 M.

## PHARMACOLOGY.

Some Compounds and Preparations of Iodine Commonly Used in Therapy. Eduardo Filippi. Ist. Stud. Sup. Florence. Arch. farm. sper., 12, 161-80.—The catalases form adsorption products with I without losing their activity. Iodized catalases do not interfere with peptic digestion in vitro or in vivo. The elimination of this adsorbed I from the organism is quite slow. Distinction between organic I compds. in which the I is attached to the central nucleus and those in which it takes the place of H in an OH group is made.

A. W. Dox.

The Physiological Action of the Chloromorphides. ERICH HARNACK AND H. HILDEBRANDT. Pharm. Inst. Halle. Arch. exp. Path. Pharm., 65, 38-53.—A certain com. prep. was found to consist of 20% apomorphine-HCl and 80% of a base which proved to be a mixt. of 2 isomers  $\alpha$ - and  $\beta$ -chloromorphide (Ach and Steinbock, C. A., 2, 415; and Schryver and Lees, J. Chem. Soc., 77, 1029) which were sepd. by fractional crystn. of their salicylates. When used therapeutically apomorphine should be free from chloromorphide. In warm and cold-blooded animals the chloromorphides exhibit in various directions an essentially accentuated morphine activity, the  $\alpha$ -base being stronger than the  $\beta$ . The effect on the breathing app. and the paralyzing action are marked. In frogs the combined paralyzing and tetanic properties of morphine are considerably stronger. The action closely resembles that of the acetyl derivs. of morphine. In its antemetic influence (in dogs) and its paralyzing action (in rabbits) it antagonizes the opposit properties of apomorphine. The therapeutic use of chloromorphide is not recommended and known cases of poisoning after apomorphine administration are attributed to its presence in the drug. WALTER A. JACOBS.

The Intestinal Action of Sulfur. THEODOR FRANKL. Pharm. Inst., Univ. Prague. Arch. exp. Path. Pharm., 65, 303-8.—The laxative action of S is explained by the fact that S is partially oxidized by the intestinal mucosa to SO, which irritates this tissue, thus producing hyperemia and increased peristalsis. A conversion of S into H<sub>2</sub>S as generally accepted was not observed.

Walter A. Jacobs.

The Physiological Action of d- and l-Tetrahydroquinaldine. DOROTHY DALE AND G. R. MINES. Proc. Physiol. Soc., 1911, xxxi; J. Physiol., 42.—Solns. of d- and l-tetrahydro-2-methylquinoline in 0.0005 M conc. act identically on the frog's heart when perfused through it. Both compds. cause diastolic arrest. This effect is attributed to the acidity of the solns. due to hydrolytic dissociation. On skeletal muscle (isolated frog's sartorius), the l-compd. produces a more rapid contraction than the d. The racemic form occupies an intermediate position between the two.

J. F. LYMAN.

Biochemical Study of Aromatic Mercury Compounds. FERDINAND BLUMENTEAL. Pathologic. Inst., Univ. Berlin. Biochem. Z., 32, 59–73.—The curative efficiency of some aromatic Hg compds. was studied. Disodium diaminodiphenylmercuridicarboxylate  $(NH_2-C_0H_2-CO_2Na)_2Hg$ , is obtained by the reduction of the compds., formed when Hg nitrobenzoate is heated to very high temp. It contains 38% Hg and does not ppt. protein. The Hg is firmly bound in the mol. and is not pptd. by H<sub>2</sub>S. This comp. may be detected by the diazo reaction and  $\alpha$ -naphthylamine. It is far less toxic than HgCl<sub>2</sub> and thus permits the introduction of larger amts. of Hg into the body. Administered per os or subcutaneously it causes no intestinal disturbances. It possesses slight anesthetic properties, subcutaneous injections are painless and are not followed by necrosis. It is rapidly eliminated in the urin. In vitro it is not antiseptic. In the animal organism it possesses spirillocidic properties. G. M. Meyer.

The Effect of Chemical Substances upon the Elimination of Sugar and upon Acidoeis. III. JULIUS BAER AND LEON BLUM. Med. klinik., Strassburg. Arch. expt. Path. Pharm., 65, 1-34.—In former articles (C. A., 1, 3018; 2, 2098) the authors showed that in dogs poisoned with phlorhizin, the subcutaneous injection of normal dicarboxylic acids with 5-8 C atoms is followed by the disappearance of glucosuria, acidosis, and high N. In the present work they sought to det. which groups of these substances were factors in this effect and what changes in the mol. would not effect this action. Completely hydrolyzed acids with 5-6 C atoms affected the sugar excretion in the same way as glutaric and adipic acids. The stereometric configuration appeared of no significance since both xylo- and arabino-trihydroxyglutaric acids behaved similarly. Mucic acid showed an anomalous behavior due to its unexpected toxicity. That the hydroxy acids are nearer to the active substance than the unoxidized is concluded from the fact that though succinic acid is inactive tartaric acid shows a glutaric acid action.  $\beta$ -Hydroxyglutaric acid must be considered as the intermediary product between glutaric and the trihydroxyglutaric acids according to its observed action and to the known course of oxidation in the organism. Symmetrical  $\alpha, \gamma$ -dihydroxyglutaric acid was likewise found to be active, the  $\beta$ -C atom being still open for oxidation.  $\alpha$ -Hydroxyglutaric acid was found to be inactive. Acetonedicarboxylic acid proved to be inactive and cannot be intermediate between hydroxyglutaric acid and the active substance. The transformation of glutaric to trihydroxyglutaric and of adipic to tetrahydroxyadipic acids is possible in the organism, that of succinic to tartaric acid impossible. The CH, group in the  $\alpha$ -position or even in  $\beta$ -position to a second carboxyl group, is not capable of oxidation in the organism (malic, tartronic and malonic acids were inactive). Former observations that in the diabetic organism OH can replace CH, and C,H, groups and that COOH in a- and  $\beta$ -positions are not replaceable by H or OH gave opportunity for confirming the above conclusions.  $\alpha, \gamma$ -Diethylglutaric (corresponding to  $\alpha, \gamma$ -dihydroxyglutaric),  $\beta$ -methylglutaric ( $\beta$ -hydroxyglutaric) removed the sugar from the urin.  $\beta$ -Carboxyglutaric acid unexpectedly showed the typical action. Glutaconic acid, apparently by addition of HOH to the double bond (forming  $\beta$ -hydroxyglutaric acid), showed marked activity. The inactivity of glutaricdiamide glutarimide showed the necessity of the COOH group. The inactivity of l-arabonic and d-gluconic and d-glucuronic acids showed that their oxidation in the organism does not occur over the dicarboxylic acids.

WALTER A. JACOBS.

The Depressor Action of the Suprarenal Glands. J. STUDZINSKI. Inst. exp. Pharm., Univ. Lemberg. Arch. exp. Path. Pharm., 65, 155-60.—The author sepd. from the suprarenal glands a fraction possessing the physiol. properties of vasodilation. To this substance he attributes the depressor action of these glands after the destruction of adrenaline.

Walter A. Jacobs.

The Influence of Phenylcinchonic Acid (Atophan) on Purine Metaboliom. EMIL. STAPKENSTEIN. Pharm. Inst., Univ. Prague. Arch. exp. Path. Phorm., 65, 177-96.—Expts. to discover how atophan increases the production of uric acid in humans gave no definit result. Working on the assumption that if the increased uric acid production were caused by an effect on purine metabolism, then in the lower mammals (dogs and rabbits) the same effect would be demonstrated by an increased elimination of allantoin (Wiechowski), the results showed a decrease in the allantoin excretion which was exactly balanced by corresponding increase in uric acid excretion. In these animals, then, the drug effects the final oxidation of uric acid to allantoin. Expts. on a rooster, which normally excretes uric acid, showed here likewise a disturbance of the purine metabolism (reduction of uric acid output) but again of a different nature from that in humans or the other mammals. In expts. on himself S. showed that under the influence of the drug no shifting of the urea N to the phosphotungstic N of the urin occurred, which speaks against the disturbance of any normally occurring oxidation of uric acid to urea (F. Frank and A. Schittenhelm, C. A., 4, 1624). Exam. of the P excreted likewise gave no clue to any increased destruction of nucleoproteins. WALTER A. JACOBS.

### MISCELLANEOUS.

Viscose (Osr, et al.). 10.

EULER, H.: Ueber blochemische Reaktionen im Licht. Berlin: R. Friedländer & Sohn. 8°, 10 pp., 6 M.

PLATT, C. AND PEARSON, W. A.: Practical Medical Chemistry. Philadelphia: J. J. McVey. 8°, 260 pp., \$2.50.

### 11b. IMMUNOCHEMISTRY.

### H. GIDEON WELLS.

Action of Solar Light on Antirabic Serum. CLAUDIO FERMI. Ist. Antirab., Univ. Sassari. Arch. jarm. sper., 12, 159-60.—Direct sunlight greatly diminishes the activity of antirabic serum.

A. W. Dox.

Anaphylaxis and Products of Proteolysis. Edgar Zunz. Univ. Brussels. Bull acad. royal méd. belg., 1911, May 27.—The three primary proteoses (heteroalbumose, protoalbumose, synalbumose) derived from fibrin are able, either directly or through the intermediation of acid albumin, to produce both active and passive anaphylaxis in guinea pigs and rabbits. Thioalbumose and the other "secondary" proteoses, fibrin-peptone  $\beta$  of Siegfried, and the totality of abiuretic substances obtained by digestion of fibrin with pepsin, trypsin and erepsin, do not cause anaphylaxis. If animals are sensitized with hetero- or proto-albumose they react with the same albumoses, entire serum, or acid albumin, but not with synalbumose, thioalbumose, secondary proteoses, or the abiuretic products of proteolysis. Animals sensitized with synalbumose react only with hetero- and proto-albumose. Reactions with albumose are less intense and slower than the usual reactions with serum. Fall of temp. is one of the most characteristic symptoms in guinea pigs. Antianaphylaxis follows recovery from intoxicating doses, and injections during this stage cause a slight rise in temp. H. G. W.

The Role of Surface Tension in the Adsorption of Toxins and Antitoxins. EDGAR ZUNZ. Univ. Brussels. Bull. acad. roy. méd. belg., 1911, Oct. 29.—Animal charcoal adsorbs diphtheria toxin and antitoxin, but not the combination of toxin with antitoxin. Talc, kaolin, BaSO<sub>4</sub>, wood charcoal and several other adsorbents will not absorb toxin, antitoxin, or the toxin-antitoxin. Animal charcoal, clay, and kaolin adsorb tetanus toxin and antitoxin, but not tetanus toxin-antitoxin. Talc adsorbs

tetanus toxin but not the antitoxin or toxin-antitoxin. BaSO<sub>4</sub> and kieselguhr adsorb tetanus antitoxin but not toxin or toxin-antitoxin. There seems to be no relation between adsorption of toxins and antitoxins and modification of the solution containing them, in regard to density, index of refraction, or freezing point. Toxins of diphtheria and tetanus seem to lower surface tension, while the antitoxins do not modify it. When toxin is adsorbed the surface tension of the solvent is increased, but adsorption of antitoxin does not always have this effect. Modification of surface tension is not a sufficient explanation of adsorption of toxins, and it seems to play no part in adsorption of antitoxins; adsorption of either is probably an electrochemical phenomenon. Union of toxin and antitoxin does not lower surface tension.

H. G. W

The Theory and Practice of Chemotherapy. Paul Ehrlich. Königl. Therap. Inst. Frankfort a/M. Folia Serol., 7, 697-714.—An address in which recent progress in the investigations on chemotherapy is discussed. Particular attention is paid to the development of strains of parasites immune to either serum antibodies or to chemical parasiticides. Serum antibodies are believed by E. to bind the same receptors as unite with foodstuffs (nutriceptors), and the parasites protect themselves by developing other groups of receptors which can only be bound by a new form of specific antibody; this counter-reaction may go on until either host or parasite succumbs. Parasites refractory to either drugs or immune bodies retain this acquired property through many generations of asexual reproductions, but lose it and revert to the original non-immune type when sexual reproduction takes place in intermediary insect hosts. Drugs which remain bound for some time in the protoplasm of the parasite cause more rapid immunization by the parasite than drugs which act and are quickly lost by the cell, independent of which drug has the more toxic effect. Organic drugs may be united to the parasite by several chemoceptors.

H. G. W.

Antitoxic Role of the Catalases. G. BILLARD. Compt. rend. soc. biol., 70, 896-8.

—Guinea pigs when injected intraperitoneally with strychnine sulfate mixed with horse serum or with egg albumin lived much longer than when injected with strychnine sulfate alone or with lettuce juice or with the juice of Amanita muscarina. This is taken to indicate that catalase exercizes an antitoxic role only when it is accompanied by a complement-like substances which is supposed to be present in the horse serum and egg albumin and absent in the juices of lettuce and Amanita muscarina.

I. K. PHELPS.

Contribution to the Study of the Precipitins. H. G. CHAPMAN. Proc. Linnean Soc., 35, 526-54; through Zentr. Biochem. Biophys., 11, 517.—Earlier work (C. A., 5, 322) is confirmed in that the ppt. is constant for a constant amt. of precipitin acting on a varying amt. of homologous protein, if only this varying amt. is greater than a constant minimum. This constant minimum is not proportional to the amt. of ppt. The latter amt. is proportional only to the amt. of antiserum. The specificity of precipitins for vegetable protein was studied and found to be of the same nature as for the proteins of animals.

# 12. FOODS.

### W. D. BIGELOW.

Report of the Activity in the Chemical Laboratory and Experiment Station of Stutt-gart in 1910. Mezger and Mueller. Pharm. Zentr., 52, 735-41, 763-8, 789-97; Chem. Ztg., 35, 1132.—The various materials analyzed (foods, medicins, com. products) are classified and the results of analyses commented upon.

A. Vorisek.

Foods. 3597

Report of the Chemical Experiment Station of Leipzig for 1910. A. RÖHRIG. Chem. Zig., 35, 953-4.—Analyses of food are given.

J. J. M.

A Study of the Touraine Milks. Eug. Desbarrières. Ann. fals., 4, 433-6.—
Desbarrières' analyses of 273 separate and 79 composit samples of milk from the Touraine region by Couturier and Salvy are given.

H. S. Bailey.

Laboratory of Good Values. M. A. Bruno. Ann. fals., 4, 430.—The history of a dispute over the comp. of milk as affected by the progress of the lacteal period.

A. W. Bromell.

The Composition of Milk. H. DROOP. Richmond. Analyst, 36, 390-2.— Tabulated results of analyses of 19,282 samples of milk. The lowest fat occurs in May and June, the highest in Oct. and Nov. The period when cows yield milk below standard appears to be extending into April and July. In general pure milk containing less than 8.5% s.-n.-f. should contain 0.5% N and 0.7% ash. The exceptions to this rule are very few.

H. S. Bailey.

The Question of Milk from Dieppe. CASSEL. Note by COUTURIER AND SALVY.

Ann. fals., 4, 436-9.—A criticism of the pamphlet by Cassel.

H. S. BAILEY.

Vegetable Milk, Vegetable Casein, and the Commercial Products Derived from the Soy Bean. Francis J. G. Beltzer. Rev. chim. ind., 22, 209-15.—The first of a series of articles on products derived from the soy bean. A description of the plant with an analysis of the fruit is followed by a detailed account of the methods of production and analysis of a fermented condiment, a milk, its conc. and desiccated forms, and a cheese known as To-Fou.

H. S. Balley.

The Chemistry of Milk and Whey Products in 1910. M. SIEGFELD. Chem. Ztg., 35, 969-71. J. J. M.

The Estimation of Total Solids in Milk by the Use of Formulas. R. H. Shaw AND C. H. ECKLES. U. S. Dept. Agr., Bur. An. Ind., Bull. 134, 1-31.—Total solids by the use of formulas of Babcock, Fleischmann, and Richmond were compared with the total solids, from the same samples of milk, detd. gravimetrically. The milk was preserved with CH<sub>2</sub>O and a composit sample for each 7 days subjected to analysis. The sp. gr. of the milk was detd. at 15° by the Westphal balance; the fat and total solids by the Babcock asbestos method (Bur. of Chem., revized, Bull. 107). In case of every cow the Babcock formula gave results closest to those obtained by grav. detn. of total solids. The Babcock formula for solids-not-fat or "plasma solids" is: plasma solids =  $\{[(100s - sf)/(100 - 1.0753 \ sf)] - 1\} \times (100 - f)$  2.5 in which s = sp. gr. of milk at 60 F and f = % of fat. 60% of 430 cases agree within 0.25 % and 90.5% of the cases agree within 0.5% of the figures obtained by grav. work. The figures calc. by the Babcock formula do not differ from the grav. in any uniform direction, but plus and minus differences nearly counterbalance; the av. diff. is only 0.07%. Eleven Quevenne and 2 N. Y. Board of Health lactometers were compared with the Westphal balance the results showing that the ordinary lactometer is unreliable for accurate work. A practical lactometer, designed to read to tenths of Quevenne degrees, was found to give results nearly identical with those of the Westphal balance. Change of temp. affects sp. gr. of milk by an amt. which varies with the total solids; the average variation per centigrade degree between 9° and 21° is 0.00025 which would account for an error of 0.08% of total solids if calc. with the Babcock formula. On taking the sp. gr. of milk after the air bubbles have escaped and again several hours later an increase is observed which begins 2-3 hrs. after milking and, if the milk be held at about 15°, continues with decreasing rapidity for 2 days. (Recknagel's phenomenon.) This change, amounting to 0.0008-0.0015 (depending on the richness of the milk) is accelerated by lower temps. and is completed in 6 hrs. at 5° or less. In making detns. to which the formula is to be applied the milk should be kept in the refrigerator for 10-12 hrs., then thoroughly mixed, heated to 60° and the sp. gr. accurately taken. Tables are furnished showing total solids in milk for any given sp. gr. and % of fat; and for total solids in 430 samples of milk, detd. gravimetrically and calc. by the formulas of Babcock, Fleischmann, and Richmond resp. L. W. Riogs.

The Action of Formaldehyds and of Mercury Oxycyanids on the Plant Growth in Milk. H. KÜHL. Suddeut. Apoth. Ztg., 51, 376-7.—Small quntities of HCOH prevent the growth of bacteria in milk but not the growth of certain molds as Peniculium glaucum, though after some time their growth is impaired. Milk preserved with HCOH showed peculiar chalk whiteness. Heated to 70° with (NH<sub>c</sub>)<sub>2</sub>SO<sub>4</sub> its casein sepd. in coarse granules, and the serum filtered clear. The casein of fresh milk began to coagulate at 80° in fine granules, and the serum filtered turbid. Since HCOH produces a change in the casein of milk, it is thought that such casein loses value as a nutrient for the mold. Milk preserved with HCOH showed a slight decrease in d., % of fat and dry residue. The fat, which evidently served as food for the mold, was strongly rancid. The action of Hg oxycyanide is like that of HCOH. 1 mg. to 25 cc. of milk showed a strong inhibitive action; 5 mg. destroyed all organic life.

A. VORISEK.

Estimation of the Bacterial Content and Testing of Milk by Recent Methods with Special Reference to Catalase Test. W. Kuntze. Zentr. Baktr. Parasitenk., II abt., 29, 616-53.—Results on bacterial content are compared with those obtained by estg. the catalytic activity of milk. The method consists of measuring the vol. of O liberated in 30 min. from 3 cc. 1% soln. of neutral H<sub>2</sub>O<sub>2</sub> by 50 cc. milk; this vol. should not exceed 2.5-3.5 cc. Such milk after 2 hrs. incubation seldom shows any increase in catalytic activity. If more than 2.5 cc. is liberated, an incubated sample should be tested. Originally high catalase may be due to colostrum, infections such as mastitis and tuberculosis, mechanical irritation, especially when injury to blood vessels allows red calls to pass into the milk, perhaps to disturbances of ennervation and other disorders such as diarrhoea. Catalytic activity is increased by high bacterial concent from any source. Catalase is closely associated with cream, but the author did not find a quant. relation between fat content and catalase. Acidity inhibits catalytic action, but does not interfere with the test so long as the milk does not coagulate with alc. G. R. HENRY.

The Electrical Conductivity of Cow's Milk. J. Th. Flohil. Chem. Weekblod., 8, 605-11.—In 122 samples the elec. cond. varied from  $45.7-54.2 \times 10^{-4}$ ; after removing 3% of fat,  $47.20-50.77 \times 10^{-4}$ ; after b. for 30 min.  $46.20-45.60 \times 10^{-4}$ . H<sub>2</sub>PO<sub>4</sub> in soln. in milk remains const. before and after b. but citric acid is diminished 30-50% depending on the length of b. The influence of spontaneous souring on the cond., when the total acidity does not exceed 32 Soxhlet-Henkel degrees may be expressed:  $A_m 10^4 = A_0 10^4 + 0.8$  ( $Z_m - Z_0$ ) where  $Z_0$  = the acidity of the fresh milk and  $Z_m$  that of the soured sample. The decrease in elec. cond. is not proportional to the degree of diln. Diluting the milk with 20% H<sub>2</sub>O does not change the cond. sufficiently to throw it outside the limit for undil. milk. About 57% of the cond. of milk is due to the presence of alkali chlorides and the variations in cond. are principally due to changes in Cl content. Such variations occur both in normal and pathological milk.

P R DINERAD

Contributions to the Knowledge of Lecithin. ROBT. COHN. Z. Offent. Chem., 17, 203-17.—The detn. of the egg content of various food materials is based upon the amt. of lecitlin present. Since the lecithin content varies from 7.5-10.5%, the detn. is not exact. Moreover, since lecithin varied in comp., the detns. based upon the content of P (detd. by ether and then hot alc. exts.) and a factor such as 11.36 or 11.34 (depend-

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ing on whether lecithin is considered as containing 2 stearic acid radicals or I of stearic acid and I of oleic acid) are uncertain. To det. whether the failure of Et.O to dissolve lecithin completely is due to combination with, or to the colloidal adsorption of the albumin present, solubility detns, were made. In the latter case the coagulating effect of the hot alc. would account for the solubility. It was found that cold alc. alone would extract practically all the lecithin and gave results closely agreeing with those obtained by the Juckenack method in which the water in the yolk of eggs is removed by calcined gypsum, the residue dissolved in ether and hot alc., the P detd. gravimetrically and multiplied by 11.36. Some expts. with the yolks of eggs, dried egg powders and lecithin preps. gave low results, probably due to the action of heat used in their manuf. upon the lecithin. Egg yolks contained about 0.9% of lecithin P<sub>2</sub>O<sub>3</sub>, Coloro, an egg powder, 1.25% and lecithin-albumin from a Berlin firm 2.3%. Euletole, supposed to be a water-sol. lecithin prep., gave roughly 100% lecithin, but actually contained none. Glycerophosphoric acid gave the necessary P content. The fraud may be recognized by the fact that the alc. extract is insol. in ether. The lipoid character of lecithin makes it doubtful that it can ever give a clear water soln., although when freshly prepared it is sol. in glycerol. To distinguish between glycerophosphoric acid and lecithin, the alc. extract should be treated with CHCl, which removes only the latter. Ether is not satisfactory as it removes also the phosphoric acid. The N to P ratio should also be 1: 1 with lecithin alone. D. S. PRATT.

Further Analysis of Ghee. E. RICHARD BOLTON AND CECIL REVIS. Analysis, 36, 392-3.—The authors tabulate the results of analyses of 7 authentic samples of ghee and point out that the R.-M. no. of 18.24, given by Kesava-Menon (C. A., 5, 1340) in view of these results and those previously given (C. A., 4, 3146) is too low. It should be about 30.

H. S. BAILEY.

Constituents of the Apple. C. THOMAE. J. prakt. Chem., 84, 247.—Preliminary paper on the odorous principle and comp. of apple peel. The peel is subjected to steam distillation and the distillate extracted with Et<sub>2</sub>O. On evapg. and treating the residue with abs. alc. a well cryst. compound is obtained. The alc. is filtered off and evapd., yielding a yellow oll of marked apple odor. By extracting the peel with Et<sub>2</sub>O in presence of dil. NaOH and evapg. there is obtained a tasteless, colorless solid, sol. in hot, insol. in cold alc., m. above 200°. This compd. is difficult to wet when shaken with H<sub>2</sub>O and forms together with plant fat or resin the waterproof covering of the apple.

P. B. Dunbar.

Biological Studies of Lemonades. J. Thöni. Bern. Zentr. Baktr. Parasitenk., II, Abt., 29, 616-43.—If in freshly prepared lemonades several thousand yeast colonies per cc. are found, there has been lack of cleanliness in manuf. Development of molds and bacteria are not favored by lemonade.

G. R. Henry.

Analysis of a Sample of Gum Drops, a New Adulterant. R. Malenfant. J. pharm. chim., 3, 484-9.—One sample consisted of a mixt. composed approx. of 25% dextrose, 25% sucrose, 35% gelatin and 15% tapioca, together with a small amt. of CaSO<sub>4</sub>.

V. K. Chesnut.

The Manganese Content of Honey. A. GOTTFRIED. Pharm. Zentr., 52, 787-8.

—In 25 samples of honey the Mn was detd. colorimetrically (Marshal method). The values obtained were 0.04-4.4 mg. per 100 g. The ash from 25 g. of a sample is taken up with hot, dil. HNO<sub>3</sub>, and the soln. transferred to a 100 cc. cylinder. The warm soln. is treated with AgNO<sub>3</sub> and (NH<sub>4</sub>)<sub>3</sub>S<sub>3</sub>O<sub>3</sub>. The red color produced is matched with a standard KMnO<sub>4</sub> soln. A certain relation to exist between the Mn content and the proportion of albumin (Lund).

A. VORISEK.

Standards for Absinthe (SCHAFFER, PHILLIPE). 16.

Test for Caramel (SMITH). 17.

Determination of Sesame Oil (KREIS). 27.

Balance of Acid-forming and Base-forming Elements in Foods (SHERMAN, GETTLER) 13.

HAGEN, F.: Untersuchungen über die Frage der Gesundheitsschädlichkeit des Eosins und der mit Eosin gefärbten Gerste. Hildesheim: A. Lax. 41 pp., 1.50 M.

JAGO, W.: The Technology of Bread-making. London: Simpkin. 21 s.

SCHERER, R.: Casein, its Preparation and Technical Utilization. 2nd Ed. New York: Van Nostrand. 196 pp., \$3.00.

# 13. NUTRITION.

WM. J. GIRS.

### NORMAL.

The Composition of Flesh after Various Diets. G. DIESSELHORST. Zootechn. Inst. Kgl. landw. Hochsch., Berlin. Arch. ges. Physiol., 140, 256-70.—From the author's expts., no general laws regarding the comp. of flesh after an abundant diet, could be deduced.

Walter A. Jacobs.

Amount of Metabolism Produced by the Breathing of Town and Country Air, and of Dry and Damp Air as Measured by the Carbon Dioxide Expired. W. Thompson. 7th Intern. Congr. Applied Chem., 1909 (Sect. VIII A), 154-60; J. Chem. Soc., 100, II, 408.

The Balance of Acid-forming and Base-forming Elements in Foods, and its Relation to Ammonia Metabolism. H. C. Sherman and A. O. Gettler. Lab. Food Chem., Columbia Univ. Proc. Soc. Exp. Biol. Med., 8, 119-20.—In continuation of previous work (C. A., 1, 2716) ash analyses have been made of a number of foods and from the % of total S, P and Cl on the one hand, and of Na, K, Ca and Mg on the other, the excess of acid over base or of base over acid which will result from the oxidation of the food has been calculated. Meats and eggs show a predominance of acid-forming elements; in fruits and vegetables the base-forming elements predominate. Milk and cereals are nearly balanced. In an expt. on a man, where the acid-forming elements of the food were increased to equiv. of 28.3 cc. N acid per day, the increased NH<sub>2</sub> excretion was equiv. to 10.7 cc. N acid per day, i. e., only \(^1/\_3\) of the extra acid was eliminated as NH<sub>2</sub> salts.

V. C. Myers.

### ABNORMAL.

Observations upon the Iron Metabolism in Pernicious Anemia and Leucemia. KLARA KENNERENECHT. Kgl. Med. Poliklinik in München. Arch. path. Anat. Virchow, 205, 89–99.—Normal urin contains about 1 mg. Fe in 24 hrs. and normal feces about 25 mg. of Fe. In anemias, especially pernicious anemia, and in leukemias, myelogenous or lymphatic, there can be an increased excretion of Fe through the urin and feces. In certain stages of pernicious anemia a decreased excretion of Fe obtains.

H. J. CORPER.

The Elimination of Salvarsan from the Human Body. FRENKEL-HEIDEN AND E. NAVASSART. Lab. der Phys. u. Nervenklinik der kgl. Charite in Berlin. Ber. klin. Wockschr., 1911, 1367-9.—The authors found no difference in the excretion of As in the urin after subcutaneous and intramuscular injections of salvarsan and obtained mirrors of 0.2-0.8 mg. for over 30 days, with a visible mirror still present after 7 months. After intravenous administration the As excretion is of shorter duration.

The excretion from the intestin was equal to and in many cases greater than that in the urin.

H. J. CORPER.

The Water Content of the Human Fatty Tissue under Different Conditions. OKTAVIUS BOZENRAAD. Med. Klinik in Strassburg. Deut. Arch. klin. Med., 103, 120-3.

—The fatty tissues of the human body have a greater water content in under-nourished individuals than in others. The av. water content in fat people was 13.2%, in emaciated persons 28.2%.

H. J. CORPER.

The Colloidal Nitrogen in the Urin in Carcinoma. STEFANO MANCINI. "R. Ospedali Riuniti" in Livorno. Deut. Arch. klin. Med., 103, 388-92.—The author concludes from his results, which agreed with those of Salkowski, that the colloidal N is increased in the urin, provided the kidney function is normal, in all disease processes in which there is an increased supply of colloid to the lymph and to the blood. This condition prevails where there is a continual resorption of exudates or transudates as in pneumonia, pleurisy, heart conditions, cirrhosis of the liver and purulent processes. In addition an increase was observed by the author in carcinoma where there may be a double origin: perverted metabolism and through autolysis of the tumor.

H. J. CORPER.

A Short Chemical Study of a Case of Cyclic Vomiting, with some Remarks on Creatinuria and Acidosis. Edward Mellanby. Physiol. Lab. of St. Thomas's Hosp. Lancet, 1911, 8-12.—The child gaving monthly attacks of vomiting, which lasted 4 or 5 days, excreted creatine during the entire period of observation. The amt. of creatine rose considerably several days before the attack. The acidosis present was only an accompanying symptom for the child's power to metabolize fats was normal. The acidosis started after the onset of symptoms and cleared up before their abatement on giving glucose, and a condition of acidosis for 4 days in a quiescent period was accompanied by no trace of ill effect. This recurrent vomiting is due to some intestinal intoxication which may or may not be bacterial in origin. H. J. Corper.

A Study of the Influence of Rice Diet and of Inanition on the Production of Multiple Neuritis of Fowls and the Bearing thereof on the Etiology of Beriberi. W. P. CHAMBER-LAIN, H. D. BLOOMBERCH AND E. D. KILBOURNE. Philipp. J. Sci., 6, 177-208.— In 56 fowls an exclusive diet of polished rice caused multiple neuritis, while those fed on unhusked rice did not acquire the disease. Those which heartily and voluntarily ate the polished rice exhibited less tendency to acquire the disease than the others. Administration of inorganic P and K salts with polished rice was without effect. No difference in effect was noted between rice with red pericarp and rice with yellowish white pericarp. Practically identical results were noted in inanition expts. and in feeding polished rice. Both K and P are markedly reduced in neuritis-producing rice and, in dietaries producing beriberi, the K more than the P. Reduction in K content seems quite as reliable as reduction in P content as an index of the power of rice to produce beriberi. No signs of multiple neuritis were apparent until there was a loss of at least 21% of the body wt.

WM. P. GARRETY.

A Contribution to the Etiology of Beriberi. W. P. CHAMBERLAIN AND E. B. VEDDER. Philipp. J. Sci., 6, 251-8.—Expts., admittedly insufficient in number, seem to indicate that the neuritis-preventing substance in rice polishings is not P, and that it is sol. in cold alc. and water, and capable of dialysis through a parchment membrane, this last consideration excluding colloids. The neuritis-preventing extract appears to have the following comp. total solids, 1.34%; ash, 0.03%; P<sub>2</sub>O<sub>4</sub>, 0.00165%; N, 0.0406%; sucrose, 0.88%.

WM. P. GARRETY.

The Behavior of Gluconic Acid and Saccharic Acid in the Organism. EDUARD SCHOTT. Med. Klinik., Strassburg. Arch. exp. Path. Pharm., 65, 35-7.—After

subcutaneous injection of Na gluconate into rabbits the author obtained only gluconic acid in the urin. This find is at variance with the results of Paul Mayer (Z. klin. Med., 47, 87) who found saccharic acid as an oxidation product of gluconic acid.

WALTER A. JACOBS.

The Concentration Function of the Kidneys. L. Lichwitz. Med. Klinik., Göttingen. Arch. exp. Path. Pharm., 65, 128-54.—From experimental material offered in a case of Diabetes insipidus and other pathological cases it was ascertained that the conc. by the kidneys of Cl and glucose is independent of that of N, and F,O<sub>s</sub>. A relation was observed between the conc. of N and that of P<sub>2</sub>O<sub>s</sub>. Diuretics were found to increase the conc. of Cl, but (with the exception of Hg<sub>2</sub>Cl<sub>2</sub>) not that of N or P<sub>2</sub>O<sub>s</sub>.

Walter A. Jacobs.

The Respiratory Exchange in Chronic Articular Rheumatism and its Modification after Muscular Exercise Provoked by Electrical Treatment. M. J. Bergonie. Compterend., 153, 129-30.—In chronic articular rheumatism the O absorbed is very much below the av. for the normal person, e. g., 331 l. during the disease as against 500 l. for a normal individual of the same wt. in 24 hrs. The ordinary methods of physical therapy, the use of light baths, etc., do not affect the respiratory exchange. Muscular exercize produced by elec. treatments increases the O consumed not only during the application (2 hrs. a day), but gradually the metabolism of O becomes normal, the musculature of the body develops and body temp. is normally regulated.

I. F. LYMAN.

The Behavior of Fat-soluble Dyes in the Organism. L. B. MENDEL AND AMY L. DANIELS. Yale Univ., New Haven, Conn. Proc. Soc. Exp. Biol. Med., 8, 126-7. The authors have studied the movements of Sudan III, under conditions where fat transport takes place (e.g., in starvation, phlorhizin and P poisoning). The dye readily migrates into the blood with the fat, but is rarely found in the liver tissue into which large quantities of fat enter (fatty infiltration). This is explained by the observation that Sudan III is abundantly excreted with the bile into the intestin from which it may be reabsorbed. Sudan III, which is insol. in H<sub>2</sub>O, is not excreted through the kidney except where alimentary lipuria is induced (in rabbits and rats). The elimination from the liver is not accomplished through the solvent medium of fat excreted in the bile (lipocholia), but the dye is sol. in bile as well as in soln. of the isolated bile salts. From an investigation of a considerable number of H<sub>2</sub>O insol. fat-sol, compds.-mostly non-toxic aniline dyes and food colors-comparable conditions were observed, pointing to the biliary secretion as a path of elimination for, fat-sol. (or bile-sol.) substances. The distribution of fat-sol. dyes within the organism depends on the presence of fat and its migrations. The dyes have not been detected in the lipoids of the nervous tissue. The authors also failed to note any inability on the part of animals to utilize fats in which Sudan III had been deposited. V. C. MYERS.

Experimental Studies on Creatine and Creatinine. W. C. Rose. Yale Univ., New Haven, Conn. Proc. Soc. Exp. Biol. Med., 8, 127-8.—The excretion of creatine induced by starvation in rabbits, is inhibited partially or completely by feeding a diet of carbohydrate alone, but is not reduced by fat alone, or fat and protein. Experimental interference with carbohydrate metabolism, e. g., phlorhizin diabetes and P poisoning, leads to the elimination of creatine. An increase in the output of creatine plus creatinine is always accompanied by an increase in total N elimination. This parallelism in inanition and with N-free diets, is ascribed to a common cause, viz., the endogenous metabolism. Coincident with the increased elimination of total creatinine during fasting, a significant increase in the creatine content of muscle occurs in rabbits and hens. Creatine is a normal constituent of the urin of the young until the age of puberty.

V. C. Myers.

The Effects of Intraperitoneal Injections of Adrenaline on the Partition of Nitrogen in the Urin of Dogs. J. ROSENBLOOM AND W. WEINBERGER. Lab. Biol. Chem., Columbia Univ. *Proc. Soc. Exp. Biol. Med.*, 8, 131-2.—Intraperitoneal injections of adrenaline chloride solns. in dogs were without effect on the proportions of urinary N in the form of CO(NH<sub>2</sub>), NH<sub>2</sub>, creatine, creatinine, purines, and allantoin.

V. C. MYERS.

ABDERHALDEN, E.: Die Bedeutung der Verdauung für den Zeilstoffwechsel im Lichte neuer Forschungen auf dem Gebiete der physiologischen Chemie. Wien: Urban & Schwarzenberg. 39 pp., 1 M.

## 14. WATER, SEWAGE AND SANITATION.

#### EDWARD BARTOW.

Analysis of the Water of San Fermin. José R. CARRACIDO. Anales soc. españ. fis. quim., 9, 213-4.—Analytical data of a chem., bacteriological and radioactive exam. of H<sub>2</sub>O from an artesian well 100 m. deep.

H. S. PAINE.

The Ozone Plant of St. Petersburg. G. ERLWEIN. Z. Elektrochem., 17, 764; Met. Chem. Eng., 9, 213-5 (4 plates).—This new plant is used for sterilizing a part of the town's water supply. The H<sub>2</sub>O from the river Neva is treated with Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> (30-40 g. per cu. m.), filtered through flint (U. S. system) and then passed into the ozonizing tank. Siemens and Halske O<sub>2</sub> generators are used. They consist of Al rod electrodes inside of cylindrical glass electrodes. The current is of 7000 v. and 500 cycles. The air is dried before entering the generators. The ozonized air (2.5 g. O<sub>3</sub> per cu. m. air) is bubbled through the filtered H<sub>2</sub>O. It is thereby freed of all cholera and typhoid germs which are very abundant in the river water. L. K. MULLER.

New Treatment of Water by Means of Aluminium Plates for Preventing Scale in Boilers without the Use of Chemicals. T. R. Duggan. J. Soc. Chem. Ind., 30, 933-4.

—Further discussion of the "Luminator" (C. A., 5, 2288). Some Al is abraded from the plates in colloidal from, which in the boiler is supposed to furnish nuclei or active centers for evolution of CO<sub>2</sub>, crystn. of salts, and combination with dissolved O, thus stopping corrosion, Milk on passing the app. was found to keep an extraordinarily long time. The discussion questioned the various theories advanced as to the ability of colloidal Al to accomplish the results claimed or of so small a current (which can not be detected), having any effect at 300° F.

D. K. French.

Treatment of Tannery Effluents. J. A. S. Morrison. J. Am. Leather Chem. Assoc., 6, 326-42.—Lime and tan liquors are mutually precipitable, so that it is necessary to run these liquids to pptn. tanks. With this lime-tannin ppt. a great part of the suspended matter, fibers, hair, dung, etc., is carried down. Either quiescent or continuous flow sedimentation is adequate. Only in special cases are chem. precipitants used. For subsequent treatment of the settled effluent the theory developed by the Mass. State B. of H. of purification by biological action in the filter beds and that of Dunbar by absorption are fully discussed. Two filters, one of graded "coke breeze" or the "first of coke" and the second of graded clinker would be efficient. For the disposal of sludge, filter presses are too expensive and simple land drainage is suggested.

Charles R. Oberfell.

Disposal of Mining Refuse. Anon. Engineering, 92, 230-1.—The pollution of streams is considered chiefly; natural causes are frequently as much to blame as industrial. The parliament of Victoria has limited to 500 grains per gal. the amt. of solid matter that can legally be discharged into any stream. Too much attention

should not be paid to discoloration of  $H_2O$ , as small amts. of suspended matter are very noticeable. D. K. Frence.

Chemistry and Hygiene. A. Röhrig. Z. angew. Chem., 24, 1619-25.

J. J. M.

Paper Water Pipes. 23.
Waste Sulfite Liquor (STUTZER). 23.
Geyser Waters in Iceland (CASARES). 8.

DARAPSKY, L.: Filterkatalyse bei der Entelsenung von Grundwässern. Leipzig: F. Leineweber. 8°, 1 M.

DIRDUI, W. J.: The Rise and Progress of Aerobic Methods of Sewage Disposal. London: The Sanitary Pub. Co.

# 15. SOILS AND FERTILIZERS.

### F. P. VEITCH.

The Significance of Soil Colloids for the Determination of Hygroscopicity in Cultivated and Forest Soils. P. Ehrenberg and H. Fick. Z. Forst. u. Jagdwesen, 43, 35-47; van Bemmelen-Gedenkboek, 1910, 194-205; Chem. Zentr., 1911, I, 1527.—Hygroscopicity detns. have generally been made on dry soils. By drying the soil, colloids must undergo great changes. Colloidal SiO, was little affected as regards it hygroscopic nature by drying while clay was significantly lessened in hygroscopicity and humus still more. In the investigation of humus and peat soils damp soils should be used.

M. X. Sullivan.

The Significance and Action of Soil Colloids on Surface Determinations According to the Rhodewald-Mitscherlich Method. PAUL EHRENBERG. Münden. Chem. Ztg., 34, 1006.—The author proposes allowing the vapor pressure of the sample to come to equil. over 10% H<sub>2</sub>SO<sub>4</sub> and then drying for the detn. By this modification the soil surface changes due to frost could be demonstrated. E. J. WITZEMANN.

Interpretations of Results Noted in Experiments upon Cereal Cropping Methods after Soil Sterilization. H. L. BOLLEY. Science, 43, 229-32.—Soil sterilization acts beneficially through destruction of disease germs rather than through its effect on the chem. properties or bacteriological activity of the soil.

WM. P. GARRETY.

Deli Soils. J. G. C. VRIENS AND S. TIJMSTRA. Meded. Deli proefstat. Medan, 5, 115-43.—Analyses of soils of the Deli region, Sumatra, show high N, poor to medium Ca, and low P<sub>2</sub>O<sub>3</sub> content. About half were low in K. Low N was found in soils having low P<sub>2</sub>O<sub>3</sub>, while high N was associated with high P<sub>2</sub>O<sub>4</sub> content. High N was found with high Ca content, and low N with low Ca content. High N was also found with low Ca, and low N with high Ca content. Also low P<sub>2</sub>O<sub>4</sub> was found with high K and low Ca content, and high P<sub>2</sub>O<sub>4</sub> with low K and high Ca content. W. P. G.

Agricultural Studies in North America with Especial Attention to Plant Breeding. K. V. RÜMKER AND E. V. TSCHERMAK. Landw. Jahrb., 39, (Ergänz. IV), 1-148.—A general review.

M. X. SULLIVAN.

Wagner Method for Determination of Soluble Phosphoric Acid in Basic Slags. W. L. Whitehouse. Bur. Chem., Bull. 137, 12.—Recommends the adoption of Wagner's method, by which the easily decomposed compds. of P<sub>3</sub>O<sub>3</sub> are dissolved out with a 2% soln. of citric acid at 17.5°. The availability results by this method agree well with those from pot expts. Data from various expt. stations prove that crops bene-

fitted nearly, or quite as much from applications of basic slag as from the ordinary high-grade phosphates.

A. W. BROOMELL.

Report of Referee on Nitrogen. C. H. Jones. Bur. Chem., Bull. 137, 14.— Lab. methods for organic N availability were investigated. The two provisional methods now in use (Bur. Chem., Bull. 107, 10–11), the neutral and alk. permanganate methods, have been slightly modified by their originators to bring the results nearer to those from pot expts. The few figures presented show good agreement between the lab. methods and the pot performances.

A. W. B.

Nitrides and their Action on Plants. A. STUTZER. Königsberg. Chem. Zig., 34, 1015.—Vegetation expts. made with Si nitride to det. whether this compd. forms NH<sub>3</sub> rapidly enough in the soil to be of value to plants showed that not enough is produced in an entire vegetation period to make Si nitride of use as a fertilizer. Other nitrides gave the same result. Mfgrs. are converting these nitrides into NH<sub>3</sub> and other available products by treating them with lye or acid under pressure. Even if nitrides become very much cheaper they would be of little value as such in the soil.

E. J. WITZEMANN.

Increasing the Yield by Fertilizing with Carbon Dioxide. P. WAGNER. Mitt. landw. Ges., 25, 176-9; through Biedermanns Zentr., 39, 726-9.—Tests on Krantz' theory that plant growth is favored by presence of large amts. of CO<sub>2</sub> in the surrounding air, the CO<sub>2</sub> coming from the fertilizer, failed to sustain it.

WM. P. GARRETY.

Manuring of Heavy Soils. And R. Z. angew. Chem., 23, 566-7; through J. Soc. Chem. Ind., 29, 504.—Excess of N manures on heavy soils reduces the yield of grain, and does not give as good results as Ca cyanamide;  $P_2O_6$  is best applied in the form of superphosphate. K salts may decrease starch content of potatoes, and give an unpleasant tasts and odor to the milk of cows fed on turnips so fertilized. Finely ground limestone is beneficial especially where K salts are liberally used. When quicklime costs but little more, this is preferable to the carbonate. W. P. G.

Manure on Chernozem. Anon. Viestnik Selsk. Khoz., 1909, Nos. 49-52; through Rass. J. exp. Landw., 11, 415.—Chernozem soils appear to require manure, the value of which depends upon the N and P<sub>2</sub>O<sub>4</sub> content. The value of K remains undetd. as does the indirect value of manure, but the after effect of manure usually lasts 4 years.

W. P. G.

The Restilization of Meadows with Potassium Salts. M. C. Dussers. Lausanne. Chem. Ztg., 34, 1040.—The author does not verify the statement of Wagner that an analysis of the ash of the plants when using K salts as fertilizer showed whether the fertilizer had been of value or not.

E. J. WITZEMANN.

Peruvian Guano. Anon. Bol. Dir. Fomento (Peru), 8, 3-4.—Analyses of 66 samples of guano from islands off the Peruvian coast showed N 1.4-14.84%, P<sub>2</sub>O<sub>4</sub> 4.6-19.45%, and K (in 9 samples) 1.39-3.9%.

WM. P. GARRETY.

The Relative Effect of Different Forms of Nitrogen on the Yield of Oats in Dependence upon the Character of the Soil and the Conditions of the Experiments. N. Ovchinnikov. Russ. J. exp. Landw., 11, 481-532.—In tests of various N fertilizers combined with a basic K and P<sub>2</sub>O<sub>5</sub> fertilizer, the largest yield was obtained in medium loam soil, the smallest in sandy soil. Light loam and clay soils were also used. Ca(NO<sub>4</sub>)<sub>2</sub>, Norwegian nitrate, and (NH)<sub>2</sub>SO<sub>4</sub> were equally efficient except that the physiological acidity of the (NH)<sub>2</sub>SO<sub>4</sub> gave unfavorable results in sandy soil. The coeff. of N assimilation was lowest for NaNO<sub>3</sub>. Steamed bone meal gave the highest coeff. of assimilation of the organic N fertilizers tested, being higher than that of NaNO<sub>3</sub>. The lowest coeff. was found with meat meal and raw bone meal, which remained practically unutilized in sandy soils. Ca cyanamide, as well as the organic N fertilizers, increased

the grain yield more than the straw. The light loam soil gave the greatest grain increase as compared with straw.

W. P. G.

Chestatee Pyrites Deposit. N. P. Pratt. Am. Fertilizer, [5] 35, 440-k.—This deposit lies in the south-east corner of Lumpkin Co., Ga. The pyrite body is from 4 1/2-40 ft. thick. The lowest grade ore so far encountered averages 51% FeS<sub>2</sub>. There is no pyrrhotite or monosulfide. The gang is a light micaceous material with little SiO<sub>2</sub>. Analysis of a composit sample from 8 carloads of lump ore gave in % H<sub>2</sub>O 0.36, S 43.52, Fe 39.70, Cu 3.09, Zn 0.72, Al<sub>2</sub>O<sub>3</sub> 2.53, MgO 0.43, As none, sand and irsol. 9.26. Practical burning tests showed satisfactory burning qualities. F. P. Veitch.

The Value of Root Residues of Various Plants as Nitrogen Collectors and Green Manure. E. Hotter, E. Herrmann and J. Stumpp. Z. landw. Versuckw., 14, 152-74.—Turning under the roots of leguminous plants produced as large an increase in yield the 1st year as did the normal application of NaNO<sub>2</sub>, and the beneficial effect of the legumes was appreciable during 2 succeeding yrs., while NaNO<sub>3</sub> had no effect the 2nd yr. The root residues of alfalfa, red clover, vetch and oats, supplied about 267.76, 156.73 and 89.25 lbs. resp. of N per acre during 4, 2 and 1 yrs. resp.; altogether more than enough to supply the need of max. crops of cereals. With rotation in which 1/5-1/6 of the total area is kept in legumes, the stubble of which is turned under, NaNO<sub>3</sub> is unnecessary. The top growth can be used as fodder for stock, the manure from which will furnish an additional supply of N.

M. X. Sullivan.

Phosphate Production in 1910. Anon. Eng. Mining J., 92, 484. J. J. M.

Chile Nitrate Industry. A. A. WINSLOW. Eng. Mining J., 92, 493.—The 158 plants in operation in the year ending June 30, 1911, produced 2,755,884 tons of NaNO<sub>2</sub> in that time.

J. J. M.

Waste Sulfite Liquor (STUTZER). 23.

Report on Potash [Determination] (BAKER). 7.

FRITSCH, J.: The Manufacture of Chemical Manures. New York: Van Nostrand 340 pp., \$4.00.

# 16. FERMENTED AND DISTILLED LIQUORS.

### ROBERT WAHL.

The Proportion of Alcohol to Extract in Wine and the Suppression of Fortifying. H ASTRUC. Revue de Viticulture, 1911; through Ann. fals., 4, 441.—The author suggests desirable changes in the standards for wines. The legal ratio between alc. and ext. of not more than 4.6 for red and 6.5 for white wines seems too low, considering improved methods of manuf. Wines less than 1 yr. old should be distinguished from older ones in regard to the above ratio.

A. W. Broomell.

The Ferment of the Bitterness of Wines the Cause of Dehydration of Glycerol. E. Voisenet Compt. rend., 153, 363-5.—The organism causing bitterness in wine is easy to cultivate, growing in mineral nutrient media containing fermentable matter. The organism is capable of fermenting various sugars and polyhydric alcs., in particular glycerol. In peptonized Laurent medium, containing 1% glycerol, the growth of the ferment is arrested by the following substances present in quantities as stated in g. per 1. Alc. 100, cream of tartar 3.5; acids: tartaric 1.1, acetic 1.2, butyric 1.0, lactic 1.6, acrylic 1.4, and Na<sub>2</sub>CO<sub>3</sub> 1.5. By the action of the ferment on glycerol acrolein is produced which when present to an extent of 0.15-0.20 g. per l. arrests further de-

velopment, but even 1 g. per l. is not sufficient to kill the organism. The acrolein is apparently formed through the action of the organism by a direct dehydration of the glycerol, there being no evidence of the formation of intermediate compds.

C. A. NOWAK.

Rothenfusser's Method for Detecting Saccharose in Wine. F. Schaffer. Berlin. Chem. Zig., 34, 1040; cf. C. A., 4, 1524; 5, 2523.—The author has found that this test depends on a reaction of methoxy furfural and that preliminary pptn. with casein and Pb(OAc), is not always necessary, but may be replaced by a distillation with 15% HCl. In the distillate methoxyfurfural may be detd. Orcinol is as good for the test as diphenylamine; after addition of HCl it gives a red with furfural and with methoxyfurfural a yellow coloration or ppt. The method has no value for wine analysis because natural wines as well as newly pressed grape juice give the reaction.

E. J. WITZEMANN.

The Investigation of and Standards for Absinthe and its Substitutes. F. Schaffer AND E. PHILLIPE. Mitt. Lebensm. Hyg., 1, 1-13; through Chem. Zentr., 1911, I, 1160. -The present usual procedure is that of Sanglé-Ferrière (Ann. chim. anal., 8, 17, 41, 127; /. pharm. chim., [6] 17, 169). The detn. of the poisonous thujone is carried out by Cuniasse (C. A., 1, 1757) according to the Legal reaction as modified by Rocques (C. A., 2, 2531) and Duparc and Monnier (C. A., 3, 764). The detn. of the ability to form a turbid mixt., according to Ackermann (Rapport d'exportise Genève, 1908), is as follows: Mix 20 cc. absinthe with H<sub>2</sub>O at 15° until a permanent cloudiness is obtained; the no. of cc. of H<sub>2</sub>O used is called the turbidity coeff. The presence of resins in addition to the ethereal oils increases the turbidity. This method is according to Duparc (Rapport d'expertise sur les imitations de l'absinthe Genève, 1908) inexact, since the different essential oils require different amts. of H<sub>2</sub>O to become turbid, and sugars and resins should be previously sepd. by distillation. The authors show that it was better to disregard the detn. of the turbidity coeff. for it is very difficult to det. the moment of beginning turbidity; it is dependent on the content of the absinthe in alc. and essential oils. The less alc. which is present with a const. content of essential oil the more readily will the turbidity ensue, and with a const. % of EtOH, the higher the content of essential oil, the greater the turbidity coeff. In order to make the Legal reaction for the detection of thujone sharper, shake after the addition of the Na nitroprusside and NaOH, wait about 1/2 min., and then add the AcOH. The Legal reaction is not obtained or only with great difficulty in absinthe or in very dil. solns. of pure thujone. By fractional distillation it is possible to conc. from solns. of high EtOH content the thujone into the last fractions which contain but little EtOH, and from solns. containing little EtOH into the first fractions. Much EtOH decreases the sensitiveness of the thujone test. In expts. with pure thujone to det. the sensitiveness of the Legal reaction, thujone was detected in dilution 1-50,000. Oils of anise, star anise and of fennel (distilled with steam) do not give the Legal reaction, whereas genuin brandies distilled from dreg lees and fermented marc of grapes did. Oil distilled from the "large" wormwood showed much stronger reaction in 1% EtOH soln. than the "small" wormwood oil. Oil of kummel in 1% EtOH soln, gave this reaction. not however in a dilution of 1-1000. The discovery of Rocques (loc. cit.) that carvone and acetone on heating with aniline phosphate, acted like thujone was confirmed Denatured spirit gives according to Legal a pronounced red color, probably as a result of the content of acetone and methyl ketone. The assumption of Duparc and Monnier that the reaction is now characteristic of thujone must be taken only with great caution A method to detect with certainty the use of the wormwood plant in a prepared liquor, is not yet available as the other plants which are usually employed for this purpose also contain thujone. The further investigations have to do with standards for absinthe and its substitutes; for these last there should be considered besides the general similarity in properties especially the essential oil content, which should be as low as possible.

R. F. BACON.

HÄGGLUND, E.: Affinitätsmessungen in alkoholischen und wässerig alkoholischen Lösungen. Berlin: R. Friedländer & Sohn. 8°, 31 pp., 1 M.

Ger., 236,486, Mar. 10, 1910. VER. DER SPIRITUS-FABR. IN DEUT., Berlin. In the exhaustive fermentation of distillery mash by means of brewer's yeast, a portion of the yeast is worked up with the mash and another portion is used to start the fermentation.

Ger., 236,591, Dec. 22, 1909. CHEM. FABR. GRIESHEIM-ELEKTRON, Frankfurt a/M. Obtaining absolute alcohol by treating spirit of over 90% with anhydrous Na<sub>2</sub>S.

# 17. PHARMACEUTICAL CHEMISTRY.

### VIRGIL COBLENTZ.

Analysis of the Extract of Licorice. L. AND J. GADAIS. Bull. soc. chim., 9, 741-3 -The following method has given satisfaction. Moisture: Dry 2 g. sample in Pt at 100° to const. wt. Insol. Material: Mix 5 g. of the ext. thoroughly with 200 cc. H<sub>2</sub>O and let stand 24 hrs. Decant, add 100 cc. H<sub>2</sub>O, decant again after standing for 24 hrs. and collect the deposit on a small weighed filter. Wash with water and dry at 100° to const. wt. Ash: Incinerate 2 g. extract in Pt. Glycyrrhizin: Dissolve 5 g. ext. in b. water. When cold make up to 50 cc. add 100 cc. alc. (95%), mix and allow to stand for 24 hrs. Decant the clear alc. liquid in a porcelain dish, collect the sediment on a dried (100°) and weighed filter and add the filtrate to liquid in porcelain dish. Wash beaker, filter and residue 3 times with 15 cc. dil. alc. (2 vol. alc. 95% + 1 vol. H<sub>2</sub>O), each time transferring the filtrate to the porc. dish. Evap. on water bath to 25 cc. and cool. Transfer to a dry weighed beaker, wash out residue in porc. dish with 25 cc. H<sub>2</sub>O, transfer to the beaker and make up to 50 cc., add 5 cc. H<sub>2</sub>O containing 1.8 HCl of 22° Bé., mix, allow to stand for 12 hrs., decant, wash ppt. and beaker 3 times with 10 cc. H<sub>2</sub>O (temp. 2°). Finally add 0.5 cc. NH<sub>4</sub>OH (d. 0.922) and dry to const. wt. at 100°. The dried residue on the filter gives the % of albuminous sub-A quicker but less accurate method is to introduce 10 g. ext. stances, gums, etc. in a flask which has 2 marks, 100 cc. and 301 cc. Add boiling H<sub>2</sub>O to dissolve, cool, make up to 100 cc., mix intimately with 170 cc. alc. (95%); after cooling add 95% alc. up to the 301 cc. mark, shake thoroughly and allow to settle. At the end of 2 hrs. filter off 150 cc. of the liquid in a porc. dish, conc. to 25 cc. and continue as in the method given above. C. BRAUBACH.

Reactions which Lead to the Formation of Iodine Derivatives. G. Gehard. Bull. sci. pharmacolog., 17, 381-2; through Chem. Zentr., 1910, II, 1050.—A soln. of 2 g. resorcinol and 1 g. I in 10 g. EtOH and 20 g. H<sub>2</sub>O, mixed with a soln. of 2 g. borax and 1 g. I in 10 g. EtOH and 20 g. H<sub>2</sub>O gives a colorless soln. which does not react with starch paste except on b. or exposure to light (when it turns red). If the soln. be conc., violet, hexagonal plates (tri-iodoresorcinol?) sol. in H<sub>2</sub>O, EtOH and EtO, crystallize out. Another colorless soln., which gives no starch-I test, is obtained by mixing a soln. containing resorcinol, I, and Na benzoate with one containing I, Na salicylate and resorcinol. If the latter be replaced by tannin, brown solns. are obtained which

give no starch-I test, and when dried at 50° yield brown sweetish scales which have but little astringency.

V. K. Chesnut.

Chemical Composition of the Rhizome of Asclepias vincetoxicum. G. Masson. Bull. sci. pharmacolog., 18, 85-9.—After extraction of the dry powder with petroleum ether, b. 60% EtOH removes a saponin-like compd., asclepiac acid, yellow amorphous, optically inactive powder, m. 90-91°, sol. in EtO, AcOR, CHCl<sub>2</sub> and AcOH, insol. in H<sub>2</sub>O. It is pptd. by BaCO<sub>2</sub> and by Pb acetate. The alkali salts are sol. in H<sub>2</sub>O, abs. EtOH and CHCl<sub>2</sub>, insol. in EtO and AcOR. The H<sub>2</sub>O solns. of alkali asclepiates are stable only in excess of alkali. The acid boiled with 5% H<sub>2</sub>SO<sub>4</sub> and EtOH in a reflux condenser splits up into a reducing sugar and an amorphous, bright red substance sol. in abs. EtOH, EtO and alkalies and insol. in H<sub>2</sub>O.

V. K. C.

New Highly Active Radium Preparations. A. FISCHER. Pharm. Post, 44, 193-4.

—Crude RaSO<sub>4</sub> is obtained directly at the Neulengbach Radium Works by a combined extraction process ("acid and alkali fusion"). The RaCl<sub>2</sub> from 10,000 kg. of pitchblende residues can thus be obtained in 6 weeks and low-grade ores containing 3-15% Ur<sub>2</sub>O<sub>3</sub>, which cannot be treated by the Curie-Debierne process are made available for use. The Neulengbach works put out preps., which in steril NaCl soln., exhibit activities of from 50-300,000 Masche units per 10 cc.

V. K. C.

The Pharmacopeial Standard for Desiccated Thyroid Glands. REID HUNT AND A. SEIDELL. Am. J. Pharm., 83, 407-11.—Hunter's method for detg. I furnishes the most reliable results and is recommended as the standard. The I should run from 0.17-0.23%, the moisture not over 6% and the ash not over 5%. Recent expts. indicate conclusively that the I is present entirely in one form and the physiological activity stands in close relation to the I content.

H. C. Fuller.

Colorimetric Test for Caramel. F. A. U. SMITH. Am. J. Pharm., 83, 411-2.

—The method consists in matching a sample of caramel against a standard color of a nesslerized soln. of NH<sub>2</sub>. (0.0417 g. monohydrated (NH<sub>4</sub>)<sub>2</sub>C<sub>2</sub>O<sub>4</sub> is dild. to 1 l., 10 cc. treated with 38 cc. H<sub>2</sub>O and 2 cc. Nessler's soln.) One g. caramel is dissolved in H<sub>2</sub>O to 1 l. and this soln. run from a buret into a Nessler glass until on diln. to 50 cc. it matches the standard. Standard caramel is considered as one of which 0.01 g. is required to match 50 cc. of the color standard.

H. C. F.

The Fixation of Sulfide by Basic Biamuth Compounds. J. L. STINGEL. Am. J. Pharm., 83, 412-3.—The suspensions of the various basic Bi salts are practically equally effective in binding H<sub>2</sub>S but in old magma this property is impaired. H<sub>2</sub>O in contact dissolves some Bi from the subnitrate and subgallate but none from the others.

H. C. F.

Standard Surgical Dressings. F. B. KILMER. Am. J. Pharm., 83, 414-24.—The rapidly changing conditions of surgical methods would not seem to warrant the insertion in the U. S. P. or N. F. of formulas for the prep. of antiseptic surgical dressings. It might be possible to establish official methods of assay by which antiseptic dressings could be judged. The standard for absorbent cotton should be revized.

H. C. F.

Some Queries on Alkaloidal Assay. W. A. Pearson. Am. J. Pharm., 83, 425-7.

—The author discusses some of the details which are of extreme importance in assaying drugs for their alkaloidal content. Crude drugs often lose 30% of moisture on grinding and the assay is then made on an entirely different product from the original, the powder is not uniform and if only particles of a certain size are taken the sample will not be representative. Temp. often influences the results; the meaning of "moderately cool," used in the U. S. P., is uncertain. Acid fumes in the lab. lower the results in titration methods. In some instances cochineal is employed and in others iodoeosin

for the same titration; it might be advizable to have a blank test run with every detn. The amt. of solvent in many instances is far too small, there are no directions for identifying the alkaloids and no physiological tests to det. the minimum lethal dose or the characteristic action.

H. C. F.

Chemical Examination of Oenanthe crocata. F. TUTIN. Pharm. J., 87, 296-8.

—The entire dried plant, consisting largely of tuberous roots, was examined. No enzyme nor alkaloid was detected. An alc. ext. yielded 3.8% sucrose, a yellow volatil oil, salicylic acid, dextrose, levulose and amorphous products; a portion of this ext. insol. in H<sub>2</sub>O consisted of a dark viscid resin from which were obtained triacontane, hentriacontane, a phytosterol, ipuranol, palmitic, linoleic and unsatd. acids and resin, the toxic portion being in the petroleum and Et<sub>2</sub>O fractions. The latter corresponds to Poehl's enanthotoxin but it is not homogeneous and is probably of complex nature.

H. C. F.

The Determination of Camphor. H. C. Fuller. U. S. Dept. Agr., Bur. Chem., Circ. 77.—The method is adapted especially to the detn. of camphor in the tincture; 25 cc. are treated in an Erlenmeyer with 2 g. NaHCO<sub>2</sub> and about 5 g. NH<sub>2</sub>OH.HCl in abs. EtOH, gently boiled under a reflux for 2 hrs., cooled to 25°, treated with 6 cc. dil. HCl, made up to 500 cc., and 50 cc. portions titrated as follows: Methyl orange is added and the mineral acid neutralized with N alkali, then phenolph. added and NH<sub>2</sub>OH.HCl titrated with 0.1 N alkali, running a blank at the same time. H. C. F.

Progress in Pharmacy. M. I. Wilbert. Washington, D. C. Am. J. Pharm., 83, 446-54.—A quarterly review of some of the more interesting literature on pharmacology and materia medica.

J. J. M.

Localization and Distribution of the Essence in Bupleurum fructicosum, Linn. L. Francesconi and E. Sernagiotto. R. ist. chim. gen. univ. Cagliari. Atti accad. Lincei, 20, II, 111-7.—Description of the distribution of the essence in Bupleurum fructicosum as observed under the microscope on fresh or stained sections of the leaf, stem, stalk, root and fruit. Several microphotographs are reproduced.

CHAS. A. ROUILLER.

Poulenc's Sodium Glycerophosphate and a Free Glycerophosphoric Acid. V PAOLINI. Ist. chim.-farm. r. univ. Roma. Atta accad. Lincei, 20, I, 807-12.—Poulenc's Na glycerophosphate corresponds in comp., within experimental errors, to the formula  $Na_2(C_2H_7O_2)PO_4$ , and gives, through the Ag salt, an acid, free from  $H_2O$  and products of hydrolysis, which forms directly, in good yield and without fractional crystn., pure  $\beta$ -brucine glycerophosphate. It seems therefore that the Poulenc salt, which is made by heating  $NaH_2PO_4$  in vacuo at  $130-80^\circ$  with 2 mols. glycerol and treating the product with  $NaOH_1$ , is practically the pure sym.  $\beta$ -salt. C. A. R.

Chemistry and Synthesis of Camphor. RUETHE. Suddeut. Apoth. Ztg., 51, 302.—Review.

A. VORISEE.

Standards for Absinthe (SCHAFFER, PHILLIPE). 16.

Knowledge of Lecithin (COHN). 12.

Rotatory Power of Camphor (MALOSSE). 2.

Physiological Action of Chloromorphides (HARNACK, HILDEBRANDT). II.

Aromatic Mercury Compounds (BLUMENTHAL). II.

Derivatives of Gelsemine (MOORE). 10.

Bulb of Buphane disticha (TUTIN). 10.

β-Camphor (BREDT). 10.

Iodine Preparations (FILIPPI). 11. Quinine Alkaloids (RABE). 10.

ASKINSON: Parfümeriefabrikation. 6 Aufl. Wien: A. Hartleben. 5.30 M.

MANNHEIM, E.: Pharmazeutische Chemie. Leipzig: Göschensche Verlagshandlung. 148 pp., 80 Pf.

Medicus, L.: Praktikum für Pharmazeuten. 3 Aufl. Tübingen: H. Laupp. 284 pp., 5 M.

Can., 134,792, Aug. 8, 1911. N. GRUNSTEIN, Frankfurt, Ger. Manufacturing acetaldehyde and condensation and polymerization products thereof from C<sub>2</sub>H<sub>2</sub>. A strong current of C<sub>2</sub>H<sub>2</sub> at 20° is passed over a mixt. of H<sub>2</sub>SO<sub>4</sub> 45% and HgSO<sub>4</sub> and products extracted by ether or the like.

Ger., 231,056, Apr. 16, 1910. Addition to 231,055; C. A., 5, 2700. FARBW. WORM. M. L. & B. In the manuf. of a tuberculosis remedy, specific tuberculosis immunizing serum is brought to act on tuberculin.

Ger., 236,080, June 11, 1910. FARBEN. VORM. F. BAYER & Co., Elberfeld. Manuf. of cholesterol preparations suitable for injection. The esters of higher fatty acids, such as olive oil, possess the property, even in small amts., of lowering the m. p. of the physiologically important cholesterol esters to such an extent that such mixts., at the body temp., m. to perfectly clear homogeneous fluids and, once molten, remain fluid for a long time at the room temp. Examples are given.

Ger., 236,154, July 12, 1908. M. RIEGEL, Berlin. A remedy for gonorrhea is obtained by extracting cultures of gonococci or B pyocyaneus, by means of a soln. contg. glucose and NaCl, at 60°.

Ger., 236,386, Aug. 19, 1910. Bad. Anilin- & Soda-Fabr., Ludwigshafen a/R. Manuf. of sulfurous acid compounds of unsaturated hydrocarbons. The hydrocarbons

with 2 conjugated double compds. of the general formula R C: CR.CR: CR

(R = H or alkyl or aryl) combined with H<sub>2</sub>SO<sub>2</sub>. These compds. are obtained by treating the hydrocarbons with H<sub>2</sub>SO<sub>3</sub> or its solns. or agents generating SO<sub>2</sub>. The hydrocarbons can be applied in the liquid or gaseous condition or dild. with suitable agents. Properties are specified.

Ger., 236,477, July 4, 1909. RÜTGERSWERKE-AKT.-GES., Berlin. Mfg. pulverulent or granular products from fusible organic substances by forcing them into the molten state under increased pressure through spraying jets and maintaining the atomized product in such state, without the aid of gaseous or vaporized agents, until the solidification results from cooling.

Ger., 236,489, May 13, 1910. A. JESSNITZER, Berlin. In the manuf. of benzoic acid from chlorinated toluene, the toluene, all of which is chlorinated in the side chain at least to such an extent as to convert it into benzyl chloride, is heated with a hypochlorite in the presence of H<sub>2</sub>O.

# 18. ACIDS, ALKALIES, SALTS AND SUNDRIES.

### T. LYNTON BRIGGS.

Reduction of Nitrosyl Sulfuric Acid with Mercury. O. WENTZKI, Frankfurt a/M. Z. angew. Chem., 24, 1468.—A reply to a criticism by Divers (C. A., 5, 2913).

The author thinks that the reduction of HSNO<sub>a</sub> by Hg takes place in two steps, thus:  $2 \text{ HSNO}_a + \text{Hg} = \text{H}_2\text{N}_3\text{S}_3\text{O}_4 + \text{HgO}$ ; then HgQ + Hg + H<sub>2</sub>SO<sub>4</sub> = Hg<sub>6</sub>SO<sub>4</sub> + H<sub>2</sub>O. He does not agree with Divers that the so-called blue acid of the lead chamber process can be formed by the reaction of HNSO<sub>a</sub> with 2 Hg, yielding Hg<sub>6</sub>SO<sub>3</sub> and HNO<sub>3</sub>, which latter products then react with another mol. of HNSO<sub>a</sub> to form Hg<sub>6</sub>SO<sub>4</sub> and H<sub>2</sub>N<sub>2</sub>SO<sub>6</sub>, but holds that this is contrary to the behavior of HNSO<sub>a</sub> toward reducing agents in general.

Edw. Wolfsensky.

German Industrial Education. C. A. TUPPER. Am. Mach., 35, 446.—In industrial and com. education the Germans are leaders. The author discusses (1) the important elements, (2) trade schools, (3) commercial education and (4) comparison of the German and U. S. systems.

C. G. F.

The Function of Technical School Laboratories. H. W. HAYWARD. Chem. Ztg., 14, 365-6. J. J. M.

Advances in the Inorganic Chemical Industries. V. HÖLBLING. Chem. Ind., 34, 443-59. J. J. M.

Activity of the Berufsgenossenschaft der Chemischen Industrie in 1910. H. Gross-MANN. Chem. Ztg., 35, 914-7, 935-6. J. M.

Atomized Water in the Lead Chambers. C. GAZEL. Technique mod., 1911, May; Ind. chim., 11, 218-20.—Description of the workings of the Pb chamber process where atomized liquid H<sub>2</sub>O is used instead of steam and of the conditions under which this process can be advantageously applied.

CHAS. A. ROUILLER.

Drying in Vacuum	(VIOLA	).	28
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Weltadressbuch der chemischen Industrie. Berlin: Union Deutsche Verlagsgesellschaft. 688 pp., 15 M.

Can., 134,932, Aug. 15, 9111. H. PAULING, Gelsenkirchen, Prussia. Process of absorbing nitrous gases. Gases are brought into contact with dil. H<sub>2</sub>SO<sub>4</sub> and afterwards condensed and dried with conc. H<sub>2</sub>SO<sub>4</sub>. They are then oxidized, subjected to wet absorbing process and again dried with conc. H<sub>2</sub>SO<sub>4</sub>.

Can., 135,070, Aug. 22, 1911. W. and K. MEFFERT, Wiesbaden, Prussia, Ger. Manufacturing zinc oxide by dissolving raw ZnO in an alkali lye, adding Zn'dust inferior to the equivalent of the foreign metals present in the soln., mixing with H<sub>2</sub>O and heating ppt. at temp. over 100° C.

Brit., 16,275, July 7, 1910. ALUMINIUM-IND., Neuhausen, Switzerland. Manuf. of hydrocyanic acid by continuous synthetic production of the same from a mixt. of

N, H and hydrocarbon, with removal of the excess of H produced by the decomp. of the hydrocarbon.

Brit., 16,455, July 9, 1910. F. A. FREETH, 45 Lawton Road, Gt. Crosby. Mfg. pure or nearly pure ammonium nitrate by causing to react, at any temp. at which they will react with formation of NH<sub>4</sub>NO<sub>2</sub>, such quantities of NaNO<sub>2</sub> and NH<sub>4</sub>HCO<sub>2</sub> (or its components) in the presence of such quantity of H<sub>2</sub>O, or of a soln. contg. NH<sub>4</sub>NO<sub>2</sub> and NaNO<sub>2</sub>, as will yield, when the reaction is finished, a ppt. of NaHCO<sub>2</sub> and a soln. satd. with respect to that salt, and also satd. or nearly satd. with respect to NH<sub>4</sub>HCO<sub>2</sub> and NH<sub>4</sub>NO<sub>2</sub> at the temp. of the reaction; removing the pptd. NaHCO<sub>3</sub> from the soln. at the temp. of the reaction and washing it to remove adherent mother liquor; treating the soln. from which the pptd. NaHCO<sub>3</sub> has been removed, in such a way as to remove the whole or the greater part of the bicarbonates, and subsequently cooling it; sepg. from the cooled soln. the NH<sub>4</sub>NO<sub>3</sub> which falls out, washing it with a satd. or partly satd. soln. of NH<sub>4</sub>NO<sub>3</sub>, and drying it by any well known means.

Brit., 17,628, July 25, 1910. J. HARGER, Grange Hollies, Gateacre. In the manuf. of oxygen, a continuous or intermittent feed of O-producing material is supplied to an igniting device in an enclosed space and the liberated hot O passed back through, around, and among the fresh O-producing materials (through a purifying device when required) to the space to be supplied with O

Brit., 24,817, Oct. 25, 1910. V. BOLLO, 42 Via Assarotti, Genoa, Italy. In the treatment of barium, strontium, potassium and sodium carbonates to obtain the resp. peroxides, the transformation of the carbonates into peroxides is effected directly by means of the catalytic action of Fe with a disengagement of gases consisting mostly of CO, at a relatively low temp. and in a relatively short space of time.

Ger., 236,341, Apr. 11, 1909. ARTIEBOLAGET SWEDISH NITRIC SYNDIKATE, Stockholm, Sweden. Continuous concentration of nitric acid from a mixt. of weak HNO<sub>2</sub> with conc. H<sub>2</sub>SO<sub>4</sub>. The acid mixt. is subjected to a two-fold heating by allowing it to trickle down in a column, tower, or tube filled with pieces of acid-proof material such as quartz, heated externally, at the same time passing upwards through the filling material hot gases which are either taken from a preheater, preferably disposed in the heating chamber of the column, or forced or sucked into this directly from the heating chamber of the column.

Ger., 236,342, Feb. 10, 1909. BAD. ANILIN- & SODA-FABR. Ammonia; see C. A., 5, 2917.

Ger., 236,373, Nov. 26, 1908. E. GERSTNER, Schöningen, Kr. Helmstedt. In the manuf. of a medium-heavy sodium chloride, from salt solns., which is lighter than the product obtained in vacuum app., the soln. is evapd. alternately in a vacuum evaporator and in an evapg. app. operating without decreased pressure, whereby the liquor obtained in the vacuum vessel, with the crystals or a part thereof, is placed in the open evapg. pan, so that crystals form which are made up of heavy and light layers.

Ger., 236,395, Feb. 19, 1910. Addition to 235,300, Feb. 19, 1910; cf. C. A., 5, 3137. BAD. ANILIN- & SODA-FABR., Ludwigshafen a/R. Mfg. ammonia. In the employment of bases the splitting off of the NH<sub>2</sub> can be materially facilitated by operating in the presence of sol. salts. In this manner, even in the presence of very small quantities of the bases, a complete transformation into NH<sub>2</sub> is effected. Further details are specified.

Ger., 236,705, Mar. 27, 1910. BAYERISCHE STICKSTOFFWEREE, Berlin. In the manuf. of ammonia from calcium nitride by means of H<sub>2</sub>O and steam, a mixt. of Ca nitride and H<sub>2</sub>O is decomposed in a counter current app. of the NH<sub>2</sub> distillation column

type by the action of steam. The nitride is completely converted into NH<sub>3</sub> even at a steam pressure of 2 to 3 atms. without the formation of dicyanodiamide.

Ger., 236,768, June 14, 1910. J. D'ANS, Darmstadt and W. FRIEDERICH, Biebesheim bei Darmstadt. Manuf. of per-acids by the interaction of peroxides with  $H_2O_2$ , employing  $H_2O_3$  in high % or  $H_2O_4$  free form. By peracids are to be understood the anhydrides of acids with per-acids. Examples are given for the manuf. of Caro acid from persulfuric acid and of peracetic acid from acetylperoxide; cf. Ber., 43, 1880.

Ger., 236,880, July 6, 1907. E. T. STEINBRECHT, Aachen-Butrscheid. Filling for reaction chambers, consisting of bodies provided on more than 2 sides with round, oval, or angular projections for the collection of flue dust. They may be cleaned and rearranged.

Ger., 236,881, Sept. 22, 1908. CHEM. WERKE VORM. DR. HEINRICH BYK. Preparations containing active oxygen; see C. A., 5, 1666.

Ger., 236,892, Oct. 21, 1909. Addition to 236,342, Feb. 10, 1909; cf. supra. Idem. Manuf. of ammonia. Instead of the Si-N compds. specified in the principal patent, the Si-N compds. obtained otherwize with the employment of N, or mixts. thereof. with decomposible N-compds. of other elements, are heated under pressure with solns. or suspensions of basic oxides, hydroxides, or salts or mixts. of these substances.

# 10. GLASS AND CERAMICS.

### G. E. BARTON, A. V. BLEININGER.

Annual Convention of the American Flint Glass Workers' Union. H. W. GAUDING. Glassworker, 29, Nos. 40, 41 and 42. G. E. BARTON.

Annual Convention of Glass Bottle Blowers' Association. David H. Jones. Glassworker, 29, Nos. 41, 42 and 43.

G. E. Barton.

Surface Tension of Molten Glasses. EDWARD WARD TILLOTSON, JR. Dept. Ind. Research, Univ. of Kansas. J. Ind. Eng. Chem., 3, 631-7 (9 figs. and 2 tables). —A variation of the drop method is developed for the detn. of surface tensions in molten glasses and a number of expts. are recorded. The results for a variety of silicate glasses vary within very narrow limits. Borate glasses have a markedly lower surface tension than the silicate glasses and the various lead borates show decided differences among themselves.

G. E. Barton.

Stoneware and Majolica. GG. C. MULLER. Glasind., 22, Nos. 29-33.—The writer discusses the firing, glazing and decorating of vessels of different varieties of stoneware and majolica.

J. B. PATCE.

The Viscosity of Glass. V. VESELY. Sprecksaal, 44, 441-3, 456-9 (5 figs.).— The viscosity at any temp. is detd. by heating to the required temp. beads of the glass between 2 hooks of Pt-Ir wire and then adding wts..which tend to pull the 2 hooks together, the time required for the operation being measured. The contact of the 2 hooks completes a circuit and deflects a galvanometer needle. The error of the measurement as conducted does not exceed 10%. Expts. with glasses I, II, III and IV show that the addition of Na<sub>2</sub>O diminishes both the m. p. and viscosity of a glass. Samples V, VI and VII show that K<sub>2</sub>O produces the same effect to a less degree. Samples VIII, IX, X, XI and XII show that the viscosity is increased by the addition of CaO, and XIII and XIV that MgO causes a much greater increase than CaO.

	SiO <sub>2</sub>	Na <sub>2</sub> O.	K <sub>2</sub> O.	Fe <sub>2</sub> O <sub>3</sub> Al <sub>2</sub> O <sub>3</sub> .	CaO.	MgO.	Heat conduc. in cm. <sup>-1</sup> g. <sup>-1</sup>	Sp. heat.	
<b>I</b>	73.29	16.09	0.90	1.38	8.04	0.22	0.002158	0.2036	
II	70.08	19.09	0.87	1.61	7.49	0.21	0.002115	0.2048	
III	67.68	21.69	0.82	1.92	7.19	0.25	0.002100	0.2074	
IV	65.36	24.53	0.95	1.52	7.06	0.23	0.002090	0.2122	
<b>v</b>	71.85	15.86	2.78	1.41	7 · 53	0.26	0.002102	0.2102	
<b>VI</b>	70.36	15.80	4.60	1.19	7.63	0.21	0.002069	0.2030	
VII	66.97	15.65	8.39	1.28	7.25	0.20	0.001991	0.1992	
VIII	68.77	15.70	0.75	1.31	13.22	0.32	0.002211	0.2036	
<b>IX</b>	65.73	15.32	0.69	1.51	16.49	0.37	0.002248	0.2038	
<b>x</b>	69.94	13.65	1.00	2.63	12.65		0.002243	0.2016	
<b>X</b> I	67.10	12.88	0.79	2.98	16.28		0.002261	0.2024	
XII	64.81	12.26	0.82	2.52	19.22		0.002283	0.2002	
XIII	68.00	13.23	0.91	2.54	11.16	4.01	0.002142	0.2031	
XIŸ	65.01	12.92	0.73	2.58	11.33	7.30	0.002107	0.2052	
	-		. •	•		. •	G. E. BARTON.		

The Injurious Influence of Sulfates in the Salt Used in the Firing of Stoneware.

MAX SCHMIDT. Sprecksaal, 44, 264.—A general discussion pointing out the defects caused by the presence of sulfates.

A. V. Bleininger.

The Influence of Metallic Oxides upon Lead Glazes. F. Kraze and A. Popoff. Sprecksaal, 44, 278-81.—The starting point of the series was a glaze of the formula RO, 0.84 Al<sub>2</sub>O<sub>3</sub>, 0.969 SiO<sub>2</sub>, containing 0.8 PbO. The balance of the RO was in turn replaced by the oxides of K, Na, Ca, Mg, Ba, Sr, Zn, Mn, Fe, Cr, Co, Ni and Cu. ZnO thus introduced was the only colorless oxide which did not show crazing on the stove tile body which was employed. B<sub>2</sub>O<sub>2</sub> did not oppose crazing. The latter defect was overcome also by the use of MnO, Fe<sub>2</sub>O<sub>3</sub>, Cr<sub>2</sub>O<sub>4</sub>, CoO and CuO. The max. fluidity was obtained by the use of CuO, followed by Co<sub>2</sub>O<sub>4</sub>, SrO, Fe<sub>2</sub>O<sub>3</sub>, BaO, MnO, K<sub>2</sub>O, Na<sub>2</sub>O, CaO, MgO, Ni<sub>2</sub>O<sub>2</sub> and Cr<sub>2</sub>O<sub>3</sub>. ZnO was not included in this series. Crazing is opposed by these oxides in the following order: Co<sub>2</sub>O<sub>4</sub>, CuO, Cr<sub>2</sub>O<sub>3</sub>, MnO, Fe<sub>2</sub>O<sub>3</sub>, ZnO, BaO, CaO, MgO, SrO, K<sub>2</sub>O, Na<sub>2</sub>O.

A. V. Bleininger.

The Preparation of some Aluminium Bisilicates. Anon. Sprecksaal, 44, 281-3, 296-8.—A review of the work of Weyberg.

A. V. Bleininger.

Interaction of Silicates and Dilute Salt Solutions. Anon. Sprechsaal, 44, 410-2.

—Review of recent work dealing with the subject of absorption and adsorption.

A. V. B.

Classification of Clays. H. Stremme. Chem. Ztg., 35, 529-31.—The following two classes of clay materials are distinguished: Feldspathic residues, grouping themselves around kaolin, Al<sub>2</sub>O<sub>2</sub>, 2 SiO<sub>2</sub>, 2 H<sub>2</sub>O, and allophane, varying between Al<sub>2</sub>O<sub>3</sub>, 0.0 3 SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>, 5.3 SiO<sub>2</sub>. To the latter class belong the zeolites which are crystallin and unstable. The feldspathic residues are difficultly, the allophanes readily, soluble in HCl. The rocks metamorphosed hydro-chemically are divided, (a), into materials occurring in situ (primary) and (b), into those of secondary origin. The former are made up of rocks decomposed by the atmosphere, by water containing CO<sub>2</sub> (kaolinized rocks) and by hot, post-volcanic vapors and gases and hot salt solutions charged with CO<sub>2</sub>. The secondary clay rocks are either produced by weathering in a temperate climate and are composed of feldspathic residue or in the tropics in which case allophane is produced, or they may be kaolinitic products, comprizing clays. A. V. B.

Phosphoric Acid Content of Ceramic Products. A. INDRA. Chem. 2tg., 35, 774.

—P<sub>2</sub>O<sub>2</sub> was found in clays, varying in amt. from 0.023 to 0.109%.

A. V. B.

Behavior of Clays towards Water. E. Donath. Chem. Ztg., 35, 774.—Confirms the statement of Cramer-Heht that dry clays break down on being brought in contact with water.

A. V. B.

BOURRY, E.: A Treatise on Ceramic Industries. A Complete Manual for Pottery, Tile and Brick Manufacturers. New York: Van Nostrand. 488 pp., \$5.00.

Brit., 23,534, Oct. 11, 1910. A. F. THOMPSON, 61 B, Fulham Park Gardens, Fulham. A combination of whiting 90, lampblack 15, paraffin oil 5, and H<sub>2</sub>O 40, mixed together and allowed to dry, forms a substance for cleaning glass.

## 20. CEMENT, AND OTHER BUILDING MATERIALS.

C. N. WILEY.

The Utilization of the Wastes of a Blast Furnace; Manufacture of Portland Cement from Slag. E. M. HAGAR. Eng. News, 66, 213. C. N. WILEY.

A Scheme for the Rapid Analysis of Cement. C. N. WILEY AND W. A. ERNST. J. Ind. Eng. Chem., 3, 551-3.—The authors make use of HOAc for the decomp. of the cement, thereby arriving at an accurate detn. of SiO<sub>2</sub> in about 20 min. Methods for the rapid detn. of MgO and SO<sub>2</sub> and the sepn. of Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> are given. A complete analysis can be accomplished in 2 1/2 hrs.

C. N. W.

Surface Tarring of Macadamized Highways. Guglielminetti. Eng. News, 66, 284-6.—The author extols the practice of using tar preparations on roads and discusses the effect of tar on vegetation.

C. N. W.

An Official German Recognition of the Harmless Nature of a Slag Addition to Portland Cement Clinker. Anon. Eng. News, 66, 291.—After an extensive exam. of the properties of the Fe-Portland cements carried out by the Royal Testing Lab., the Prussian Minister of Public Works has issued the decision that Fe-Portland cement, specified as containing up to 30% of subsequently added ground slag and Portland cements are to be considered of equal value, and insofar as the former conforms to the standards laid down for true Portland cement, it may be used on all public work. It is prescribed, however, that all such cement must be labelled "Eisenportlandcement."

C. N. W.

The Different Iron and Slag Cements. Ed. Eng. News, 66, 294.—The editor calls attention to the 4 classes of these cements. There are (1) a high-Fe cement, a species of Portland with high-Fe content; (2) a slag cement which is merely a mixt. of unburned material; (3) a true Portland mixed after burning with a pulverized slag, known abroad as "Eisenportlandcement"; and (4) a true Portland in which slag is used as one of the basic ingredients.

C. N. W.

A Prepared Filler for Macadam Roads. CHARLES H. HOYT. Eng. News, 66, 198-9.—The prepared filler is made by mixing the stone chips or pea gravel and the sand and bituminous binder. This filler is then distributed over the top course of stone and afterward properly incorporated with it.

C. N. W.

The Pressure of Wet Concrete against Forms. S. Tomomatsu. Eng. News, 66, 211.—The author has expressed the hydrostatic pressure of "sloppy" concrete against forms with a formula which he explains by means of a plot.

C. N. W.

Magnesia Cement. Anon. Industria, 25, 20; Ind. chim., 11, 234-5.—Brief summary of our knowledge as to Sorel cement.

C. A. ROULLER.

Some Notes on the Setting of Cement. W. A. B. WALLING. Chem. News, 104, 54.—In Thorp's Dic. of Applied Chem., 1, 477, giving the analysis of a cement from Heidelburg containing CaO 44.22 and MgO 17.77%, the statement is made that a good cement burned at high temp. corresponds to 100 (SiO<sub>2</sub>: R<sub>2</sub>O<sub>2</sub>) 304 (CaO.MgO) and the theory given that at a low temp. (400°) the MgO is causticized and acts as an hydraulic agent while at higher temps. both CaO and MgO silicates act as hydraulic agents. The burning and micro. exam. by the author of the 1-1, 2-1, 3-1, 1-2, 2-3 and 4-3 Mg silicates showed the presence of plate crystals in all but the 3 MgO.SiO<sub>2</sub>. None of them set on hydrating with water and it was concluded that the strength of the Heidelburg cement was due to the Ca silicate, the Mg playing no part except to weaken the cement.

A. I. Phillips.

Power and Heat Distribution in Cement Mills. H. STRUCKMANN, et al. J. Am. Soc. Mech. Eng., 33, 1024-8.—A discussion of a paper by Landis (C. A., 5, 2317).

A. I. PHILLIPS.

Effect of Lime in Cement on Durability of Concrete. H. SPACKMAN. Shop Notes Quarterly, II, 557-8.—Attention is called to the disintegration of cement clinker on storing, the retrogression in strength of briquets, the fact that only a part of cement is completely hydrated and its decomp. by alk. and sea water as showing that too much lime is present.

A. J. P.

Some Building and Ornamental Stones of New South Wales. R. T. BAKER. J. Proc. Roy. Soc. N. S. W., 43, 180-97. J. J. M.

Service Test of Road Materials by the Road Board of Great Britain and a Description of Various English Proprietary Pavements. Anon. Eng. Contracting, 36, 227-32.

—In an experimental road the materials to be used are listed. Various claims as to special properties of the compds. are made and cost of laying is given.

A. J. PHILLIPS.

Trinidad Asphaltic Petroleum and its Use as Road Material. A. SOMMER. Record, 64, 24-6.—Well sinking on the borders of Pitch Lake, Trinidad, yielded very heavy oils, viscous at ord. temp. and very suddenly changing their viscosity at higher temp. Fractionation showed that the distillate is chiefly naphtha, heavy intermediate, fractions and higher-b. constituents being absent. The material is a crude maltha yielding about 5% of asphaltenes on treatment with petroleum ether and is well adapted for road use. The 2 classes of soft asphalt now on the market are: (1) Paraffin or semiparaffin residuals owing their consistency to a large % of viscous paraffin hydrocarbons and (2) solid asphaltic bitumen products. Asphalts for road purposes should possess both adhesiveness and elasticity. The paraffin oils lack the former since by exposure to weather and the action of capillarity the lighter oils will rize to the road surface, the heavy oils follow more slowly and the mineral aggregate will retain the asphaltic comp., resulting in slippery roads in wet weather (cf. C. A., 5, 2549). "Bleeding" of a road is always due to the presence of paraffin hydrocarbons in the oil. The liquid asphalts can not be applied to dusty roads as they form lumps with the dust; they do not spread like paraffin oils but remain where applied, and from them asphalts of considerable solidity can be obtained by distillation at low temp. Any disintegration or loss of adhesive power by cracking is thus obviated (C. A., 4, 2041).

A New Consistemeter for Testing Bituminous Road Materials. W. W. CROSBY. Good Roads, 13, 116-8.—The app. consists of a balanced wheel, grooved at the circumference and provided with a measuring device to det. the arc through which the wheel is turned. In this way is measured the distance traveled by a disk of known area and load, through the substance contained in a box of fixed dimensions in a fixed

interval of time. A record of results and some comparisons with the petrometer are given.

A. J. P.

Brit., 16,028, July 5, 1910. H. J. Harms, Jr., 50 Carey St., Orange, N. J. In the manuf. of liquid concrete, a mixt. of clay and H<sub>2</sub>O is added to cement, sand and fine stones, the clay being in a finer state of division than the cement, and remaining floating in H<sub>2</sub>O, the proportion of clay being exactly detd. by means of a hydrometer.

Ger., 235,756, Feb. 21, 1908. A, LEVY, Amsterdam. In the manuf. of coating masses which contain sorel cement as binding agent, the magnesite serving for the production of the binding agent is incompletely burned, still containing the carbonate.

# 21. FUELS, GAS AND COKE.

### J. D. PENNOCK.

Nature of Some Coal Dusts and Mine Air from Mines in Colorado. J. B. EKKLEY.

J. Ind. Eng. Chem., 3, 586-8.—Data consisting of coal dust analyses and mine air analyses from some of the more important mines of Colorado.

C. A. Coles.

Determination of Sulfur in Coal by Means of Jackson's Turbidimeter. H. F. Murr. J. Ind. Eng. Chem., 3, 553-7.—An investigation to detn. the causes of variations between grav. results and those obtained by the turbidimeter. Variations of size and distance of flame from the soln. and small quantities of nitrates do not affect results appreciably. Differences in size of BaCl<sub>2</sub> crystals used in pptn. of SO<sub>2</sub> and large excess of HCl are chief sources of error. By using uniform BaCl<sub>2</sub> crystals pressed into tablets of definit wt., the chief difficulty is obviated.

C. A. Cole.

Accurate Estimation of the Calorific Value of Anthracite Coal. G. BLAKELEY AND E. M. CHANCE. J. Ind. Eng. Chem., 3, 557-9.—Chilling the coal by rapid conduction of heat through the Ni capsule is the real cause of incomplete combustion in the bomb calorimeter. By lightly tamping into the bottom of the capsule 3 mm. of previously ignited asbestos, perfect insulation was secured and hard coals with as much as 40% ash were completely burned.

C. A. C.

The Reaction Attending the Coking of Coal. R. F. CARPENTER AND S. E. LINDER. J. Gasbel, 54, 253-5; Chem. Zentr., 1911, I, 1531.—Lab. expts. on the interaction of coal gas and NH<sub>2</sub> or HCN at various temps. seem to indicate that NH<sub>2</sub> is converted partially into HCN by the decomp. of CH<sub>4</sub> and C<sub>2</sub>H<sub>4</sub>. Definit mixts. of coal gas and NH<sub>3</sub> or HCN were conducted through a porcelain tube filled with charcoal, coke or graphite at temps. ranging from 850 to 1100°. HCN in presence of coal gas does not decompose appreciably below 1000°, but above that temp. the decomp. is rapid. In presence of coal gas less NH<sub>2</sub> is decomposed into its elements and more HCN formed, undoubtedly through the decomp. of CH<sub>4</sub>. Presence of C<sub>2</sub>H<sub>4</sub> furthers the formation of HCN from HN<sub>3</sub>. This reaction is reversible, the equil. depending on the temp. A number of tables give the detailed results in each expt.

G. N. Terziev.

Flow of Heat through Furnace Walls. C. Hering. Met. Chem. Eng., 9, 438.—Comment on the article of Ray and Kreisinger; C. A.; 5, 3508.

J. J. M.

The Relation of Illuminating Gas to Public Health. W. T. SEDGWICK AND F. SCHNEIDER. Eng. News, 66, 314.

J. J. M.

Synthetic Production of Methane. E. ERDMANN. Chem. Trade  $J_1$ , 49, 175.— The author has succeeded in working out a successful com. process of converting water gas into  $CH_4$  as follows:  $CO + 3H_2 = CH_4 + H_2O$ . In practice it is necessary to use 5 vols. of  $H_2O$  to 1 of CO. Water gas contains 52 of H to 40 of CO. The process in brief: The water gas is freed from tar and subjected to a pressure of 10 atms. to free it from CO<sub>2</sub>; the remaining 0.1% CO<sub>2</sub> is removed by lime. The Linde machine then partly liquefies the CO (used to drive compressors); by expansion valves, the % of CO remaining in the gas can be controlled within 1%. The issuing gas has the comp. 17% CO, 79% H and 4% N; it is reduced by passing over finely divided Ni in SiO<sub>2</sub> tubes at 300°. In removing the excess of CO,  $H_2$ S (detrimental to the activity of the catalyzer) is also removed. The activity of the Ni, which is impaired by the deposition of C, can be completely restored by passing over it gas weak in CO. A gas having the comp.: 40% CO, 4% CO<sub>2</sub>, 52% H and 4% N and cal. value of 2550 cals., after going through the process contained traces of CO and CO<sub>2</sub>, 30% CH<sub>4</sub>, 62% H and 6-7% N. The total cost for English conditions for a gas of 32% CH<sub>4</sub> is 8.5d per 1000 ft.

G. N. Terziev.

An Asbestos Unit of Light. R. A. HOUSTON. J. Gas Lighting, 115, 412. —When a light filter capable of stopping all the infrared and ultraviolet radiation and of reducing the energy of the visible spectrum in inverse ratio to its light-producing effect is placed in front of a thermopile, the radiation will be measured according to the visibility, and the deflection will be proportional to the light received. Such an app. can be used to measure c. p. and especially mean spherical c. p. The unit of light intensity is that source, the total intensity of radiation from which, at an optical distance of 1 m., after passing through an ideal filter, would be x ergs per sq. cm. per sec.; the ideal filter possesses the light-absorbing properties of 3 cm. of an aq. soln. of cryst. CuSO<sub>4</sub> (0.2 g.-mol. per l.) and 1 cm. of an aq. soln. of  $K_2Cr_4O_7$  (0.0025 g.-mol. per l.); but neither to reflect nor absorb any light in any other way. The value of x for stand, candle in the units specified is roughly 0.8.

M. B. SMITH.

Gas Testing and Illuminating Power in Canada. Anon. J. Gas Lighting, 115, 413.—The Canadian Gov. has adopted the No. 2 "Metropolitan" burner for testing the illuminating power of gas, the new burner replacing the Sugg standard burner. It was further recommended to reduce the c. p. of gas to 14 and the B. t. u. to a minimum of 450 net.

M. B. S.

Manufacture of Ammonia in By-product Coke Ovens. L. C. Jones. J. Ind. Eng. Chem., 3, 589-94.—A discussion of N in by-product coke oven operation and the recovery in the form of NH<sub>2</sub> as affected by variations in coke oven operation.

C. A. COLE.

Coking Industry of South Yorkshire and Derbyshire. L. T. O'Shea. J. Soc. Chem. Ind., 30, 937-40.—A general review of the coking industry of the above-named districts and discussion of various methods of by-product recovery. C. A. Cole.

By-product Ovens in America and Europe. F. E. Lucas. Iron Age, 86, 375-6.
L. A. Touzalin.

Gas Lamp Ignition (PIERCE). 1.
Recovery of Benzole (BAGLEY). 22.

BRÄHMER, F.: Chemie der Gase. Frankfort a/M: F. B. Auffarth. 6 M.

Ger., 235,870, Sept. 28, 1909. K. BURKHEISER. Removal of hydrogen sulfide from gases; see C. A., 5, 3148.

Ger., 236,757, July 3, 1909. K. BURKHRISER. Ammonium sulfite or ammonium sulfate; see C. A., 5, 3335.



# 22. PETROLEUM, ASPHALT, COAL TAR AND WOOD PRODUCTS.

### R. E. HUMPHREYS.

Petroleums and their Industries. FABIO VILLANI. Ind. chim., 11, 165-9, 197-200, 213-8.—Review of the mode of occurrence and of the methods of working up petroleums.

Chas. A. Rouiller.

Fuel Oil Specifications. I. C. ALLEN. Eng. Mining J., 92, 582.—The U. S. Bur. of Mines has issued specifications for the purchase of fuel oil with directions for sampling oil and natural gas. Fuel oil should be either a natural homogeneous oil or a residue from a natural oil. The flash point should not be below 60° in a closed Abel-Pensky or Pensky-Martens tester. The d. should range from 0.85-0.96 at 15°. The oil should be mobil, free from solids or semi-solids and should flow readily at ordinary temps. and under a head of 1 ft. of oil, through a 4-in. pipe 10 ft. in length. The calorific value should be not less than 18,000 B. t. u. per lb. It should be rejected if it contains more than 2% H<sub>2</sub>O; more than 1% S, or more than a trace of sand, clay or dirt.

Manufacture of White Vaselin Oils. F. KRAJENSKI. Seifensieder Zig., 38, 539, 581.—Mineral oils with a naphthene base, also Russian oils are best suited for making white vaselin oils. Mineral oils with a paraffin structure are not suitable on account of poor viscosity.

E. Scherubel.

The Recovery of Benzole from Coke-oven Gases. D. C. BAGLEY. *Iron Coal Trades Rev.*, 83, 235.—A detailed and illustrated description of an installation of the "Still" system of benzole recovery and rectification. The uses of the different products in the arts are chiefly in aniline dye and explosives manuf., for gas enrichment, as solvents in the rubber and varnish industries. As motor spirit in internal combustion engins ninety % benzole is the material used almost exclusively in Germany and very largely in France. Tests on a 40 h. p. 4 cylinder motor showed that with proper carburetting methods the quantity of benzole used was 20% less than that of 0.740 petrole for a given output. As an economic factor in the production of coke the recovery and rectification of benzole plays an important part.

Trinidad Asphaltic Petroleum (SOMMER). 20. Smelting with Oil (JACOBS). 9. Purification of Acetic Acid (ORTON). 10.

RAKUSIN, M.: Die Polarimetrie der Erdöle. Berlin: Verlag für Fachliteratur. 12 M.

Ger., 235,777, Oct. 8, 1910. M. SINGER, Budapest; LEOPOLD SINGER, Pardubitz, PHILIPP PORGES, Vienna and RICHARD NEUMANN, Königsfeld bei Brunn. App. for separating oil from paraffin and fractionally m. out the paraffin by means of circulating warm H<sub>2</sub>O in perforated containers arranged therein, consisting of a warm H<sub>2</sub>O container which is divided by intermediate walls into 2 portions or groups of such containers, of which the 1 receives the paraffin container formed of pierced or sieve-like walls, while in the other the circulation app. is disposed, which mixes the warm H<sub>2</sub>O and divides it uniformly through openings of the intermediate walls and leads it with equal temp. to the paraffin mass in the containers so that this is uniformly washed.

Ger., 236,050, Oct. 8, 1909. J. TANNE and GUSTAV OBERLÄNDER. Separation of solid hydrocarbons; see C. A., 5, 3340; Brit., 23,125.

Ger., 236,051, Oct. 28, 1909. Addition to 236,050, Oct. 8, 1909; cf. supra. Idem. In the separation of solid hydrocarbons, such as paraffin, ceresin, or ozokerite, from petroleum residues and tars, the halogen substitution products of the satd. and unsatd. hydrocarbons, such as dichloroethylene, are employed instead of CCl<sub>4</sub>.

Ger., 236,199, July 10, 1908. J. POLIFKA and B. HACKER, Budapest. A wood impregnating compound consisting of a wood tar oil contg. 40-70% creosote, and a beavy or high-b. mineral oil distillate which serves as solvent for the wood tar oil. The wood tar oil can be replaced by coal tar oil contg. carbolic acid.

Ger., 236,340, Oct. 8, 1909. A. BERTELS, Hamburg. Restoring wood charcoal for filtering by passing therethrough combustion gases which contain little or no free O or reducible O compds. and which contain sufficient heat to drive off vol. impurities. These gases can be generated in a sep. generator, and by means of a special construction of the generating container for the charcoal an uninterrupted process is rendered possible. Without continuity of the process, the combustion gases can be generated directly in the regenerating container.

# 23. CELLULOSE AND PAPER.

#### A. D. LITTLE.

Paper Water Pipes. Anon. Papier-fabrikant, 9, 604.—The water pipes in an ancient convent at Dürkheim were found to be made of paper with intermediate layers of asphalt. The walls of the pipes are 20 mm. thick and consist of 20 layers of paper. Microscopic exam. showed cotton and wool.

V. Nunez.

Metallic Iron in Paper. Anon. Papier-Fabr., 9, 633.—In rag papers, Fe is introduced through buttons, hooks, etc. In wood pulps, straw-cellulose, etc., the Fe probably comes from the scraping of the knives of the beater-roll against the bed plate. The Fe may be in great part removed by use of magnetic rakes. V. Nunez.

Sugar-cane Paper. Anon. Paper Zig., 36, 2221-2.—A discussion of the chance, which paper made from bagasse has commercially in competition with wood-pulp paper.

V. Nunez.

Strength of Paper. How it is Influenced by its Weight. Arnold Rehn. Paper Mill, 34, 15; Papier-Fabr., 9, 973-6.—Inasmuch as the influence of the thickness of a paper upon its strength is very great for a paper of more than standard wt. and yet of high strength, much better finish than usual is necessary. In making abnormally thin paper, difficulty is had in obtaining the required folding resistance, since extreme care must be taken in the beating and machine work. There is a certain thickness of paper for every material, at which the max. breaking length is attained, and it may be advizable in the case of a paper of abnormal wt. not to maintain the values of strength which are fixed for paper with a standard wt.

V. Nunez.

Raw Paper for Art Prints. Anon. Papier-Fabr., 9, 880-1. J. J. M. History of Paper. J. von Wiesner. Papier-Fabr., 9, 886-7. J. J. M.

The Output of Grinders. G. KEIL. Papier-Fabr., 9, 912-3. J. J. M.

The Throwing-sorting Machine or the Centrifugal-sorting Machine? Anon. Papier-Fabr., 9, 919-21, 47-8.

J. J. M.

The Chloride Caustic-soda Plant in the Pötschmithle Cellulose Works. W. EBERT. Papier-Fabr., 9, 1002-10. J. M.

New Japanese Paper Mill at Hokkaido. Papier Zig., 36, 2293-4, 2330-1.

V. Nunez.

Use of Waste Sulfite Liquor as Irrigation Water. STUTZER. Königsberg. Wockbl. Papierfabr., 42, 2685.—A sulfite pulp mill, above Königsberg, was allowed to discharge its waste liquor into the city irrigation canal, provided the following conditions were complied with: (1) the waste liquor should contain not more than 0.06% of free acid (2) the temp. should not exceed 40°, (3) the rate of discharge should be uniform. The total daily discharge of the canal was 26,000 cu. m. of which the sulfite mill contributed 11,000 cu. m. The Fe work of the canal was not seriously attacked but growing plants were injured by the free acid. The neutral sulfite salts and the organic matter were not deleterious. When the free acid did not exceed 0.02%, no injury to plants occurred and a certain improvement in the crops was noticed. V. Nunez.

Formic Acid and Cellulose (CROSS, BEVAN). 10.

BEVERIDGE, J.: Paper Makers' Pocket Book. 2nd Ed. New York: Van Nostrand. 8°, 211 pp., \$4.00.

CROSS, C. F., BEVAN, E. J. AND SINDALL, R. W.: Wood Pulp and its Uses. New York: Van Nostrand. 281 pp., \$2.00.

DALEN: Chemische Technologie des Papiers. Leipzig: J. A. Barth. 4 M.

Brit., 15,991, July 5, 1910. H. BERNSTEIN, 1019 Race St., Philadelphia, Pa. Mfg. cuprammonium solution by subjecting metallic Cu to a mixt. or soln. of aq. NH<sub>a</sub> and molasses, and passing a current of air or any suitable gas contg. free O through the liquid.

Brit., 29,087, Dec. 14, 1910. J. C. W. STANLEY, 246 Riverside Ave., Santa Cruz, California. Production of wood pulp from woods of a resinous nature by soaking the splintered wood in hot NaOH soln., straining from the liquor, crushing, washing, and passing through a press.

Ger., 235,965, Aug. 25, 1910. Addition to 220,066, Dec. 16. 1908; cf. C. A., 4, 2203. A. MITSCHERLICH, Freiburg i/B. In the manuf. of emulsions suitable for the preparation of paper sizing, horn soln. mixed with crude sulfite cellulose waste lyes is pptd. with HCl, the ppt., sepd. from the soln., is dissolved in H<sub>2</sub>BO<sub>2</sub>, and the soln. is mixed with an alk. resin soln., with a view to obtaining emulsions wholly or almost free from Fe. By the addition of dehydrating salts, a finishing size in solid form is obtained.

Ger., 236,035, Dec. 8, 1909. A. STUTZER, Königsberg i/Pr. In the removal of the sulfurous acid from the waste lye of the sulfite-cellulose manufacture, the hot waste lye is made alkaline by the addition of NH<sub>4</sub>OH or (NH<sub>4</sub>)<sub>2</sub>CO<sub>2</sub>, the liquid is filtered after a time or otherwise clarified, conc. by evapn., again filtered, and further evapd. to a sirupy or solid mass.

# 24. EXPLOSIVES.

### C. B. MUNROE.

Experiments on Liquid Mixtures for Laying Coal Dust. W. M. THORNTON. Coll. Guard., 102, 273, 318.—Great colliery explosions, however they may originate, are spread by the progressive ignition of coal dust. To combat coal dust we may (1) reduce its formation, (2) remove it wherever found, (3) fix it so that it cannot be raised in a cloud, and (4) dil. it with stone dust to render it inert. Probably a combination of the four methods will ultimately be found best. Watering is not efficacious for

fixing coal dust, possibly because of the high surface tension of H<sub>2</sub>O. Liquid mixts. of lower surface tension than water and with a slight solvent action on the coal possess wetting powers much greater than water. The best mixt. in respect to both efficiency and cost was one of water glass 10, resinous liquid soap 1, and com. PhOH 1 (5% solns.). PhOH may be dispensed with if the proportion of soap is increased. The water glass acts as a binder and hardener of the dust, rendering it less inflammable and counteracting the effect of the increase in volatil matter caused by the soap. The latter effect is of little importance since the quantity of soap added is only about 3% of the wt. of coal dust. A table of surface tension values and wetting powers of different liquids shows that the latter value is dependent somewhat upon other properties as well as surface tension, probably solvent action on the coal for the most part. Actual trials in a mine showed that the method of spraying must be very thorough to obtain good results and that thick deposits of dust can be effectually dealt with only by removal.

WORDEN, E. C.: Mitrocellulose Industry. New York: Van Nostrand. 1239 pp., 2 Vols., \$10.00.

Brit., 15,925, July 4, 1910. C. STUART-BAILEY, 67 Westbourne Road, Lancaster. Preventing or minimizing explosions of coal dust in coal mines by scattering through air in the mine a fine powder, as a result of an initial explosion acting on a suitable store or stores of the powder which consists in arranging in the roadways and galleries, as also at the working coal faces of the mines, and in conjunction with wedges as tamping in the bore hole, hermetically sealed packages of waterproof and incombustible material charged with the fine powder in anhydrous condition and so constructed that they are burst as a result of the initial explosion or the operation of tamping and the powder is scattered.

Brit., 21,071, Sept. 10, 1910. A. C. LANFREY, Baccarat, France. An explosive formed from CaCO<sub>2</sub> 1.5, picric acid 3, powdered KNO<sub>2</sub> 5.5, powdered C 1, and NH<sub>2</sub> 1.

Brit., 26,334, Nov. 12, 1910. NITROGLYCERIN ARTIEBOLAGET, 9 Arsenalsgatan Stockholm, Sweden. A plastic explosive having as basic material the so-called liquid di- or trinitrotoluene of commerce, with or without the addition of other substances dissolved therein, gelatinized by means of nitrocellulose, this basic material being mixed with finely powdered O-yielding substances such as NH<sub>4</sub>ClO<sub>4</sub>, alk. nitrates, and the like.

## 25. DYES AND TEXTIL CHEMISTRY.

### L. A. OLNEY.

The Bulgarian Textil Industry. Textil Faerb. Ztg., 9, 152.—The progress of the native industries in linen, jute and hemp.

JOSEPH HAROLD.

Hydron Blue. G. AND R. B. WEILL. Leipziger Farber Ztg., 60, 275-8.—These new vat dyes, which are condensation products of carbazole with p-nitrosophenol are superior to indigo in the light, Cl, soaping and rubbing tests. The leuco compds. are level dyeing. Recipes for dyeing are given.

A. S. HALLAND.

Technical Education—Its Function in Training for the Textil Industry. C. H. RAMES. Chem. Eng., 14, 363-5.

J. J. M.

Brocade Effects on Cotton Goods. Textil Fastb. Ztg., 9, 185-6.—Fabrics, after mercerization and while still damp, if pressed with a roll bearing a design in relief,

are found to have a variable absorptivity for liquids, the stamped parts taking less dye or mordant. There is an addition to the two-color effect thus rendered possible, a variation in the gloss of the pressed and unpressed part and a relief effect.

JOSEPH HAROLD.

New Mercerizing Machine. Textil Faerb. Zig., 9, 186-7.—Spool carriers are arranged radially about a common center so that they may be rotated in a horizontal plane during the flow of the mercerizing liquor and wash waters through the yarn.

JOSEPH HAROLD.

New Yarn-dyeing Machine. Textil Faerb. Ztg., 9, 221-3.—The advantage of the machine is that the yarn is held in such a way, that it may be lifted and passed, while in constant motion, through a series of baths such as are employed in the operations of mordanting, dyeing and washing, without demounting.

JOSEPH HAROLD.

Brightening Piece-dyed Turkey-red Goods. Textil Faerb. Ztg., 9, 233-5.—The app. is designed to brighten the goods by the froth process but works in an open kettle, the fabric passing over squeezing rolls in and out of the froth. JOSEPH HAROLD.

Electrical Installations in Cotton Print Works. L. Kuhl. Textil Faerb. Ztg., 9, 245-8, 257-9, 269-72; Z. Farben-Ind., 10, 83-8, 99-102.

Notes on Dimethylaniline and its Derivatives. I. Johann Walter. Geneva. Z. Farben-Ind., 9, 378.—Investigations on the flash-point of dimethylaniline and the effect of its vapors on workmen. Its reaction products with  $\beta$ -naphthaldehyde and with sugar are discussed and the history of the production of sun-yellow is given. The coloration given by dimethylaniline to wood fiber and subsequent reactions of the color are critically considered. II. Ibid., 10, 17–20, 33–5. The methods of making tetramethyldiaminodiphenylmethane are considered and the part played by the O of the air in its derivation from dimethylaniline is discussed although expts. here detailed show no definit product from dimethylaniline on long exposure to air in shallow dishes. The effect of air on it at elevated temps. in the presence of AcOH is studied, the absorption of O by the mixt. being quant. observed. The author aims to make clear the process of violet production which, despite the considerable investigations of E. and O. Fischer, is still somewhat obscure.

Cotton Production in Egypt. Textil Faerb. Ztg., 9, 259.—A new variety "Sakelaridi" has been found to surpass other plantings, and this year it has been much cultivated.

IOSEPH HAROLD.

The Color Reactions between Aromatic Amines and Nitro Compounds. JOHANN WALTER. Geneva. Z. Farben-Ind., 10, 49-51, 65-8.—The author investigates anew the colorations given by a very large number of amines with nitro compds.

J. H.

The Compounds of the Basic Dyestuffs with Tannin. A. Sanin. Lab. Kiev Polytechn. Z. Farben-Ind., 10, 81-3, 97-9.—A development of the author's earlier researches on the tannin and tartar emetic reaction. Pure cryst. chrysoidine, fuchsin, crystal violet, brilliant green and Victoria blue were employed with a very pure tannin of known strength. Equiv. mol. solns. of dyestuff and tannin were prepared and pptn. effected by NaOAc. The tannic acid liberated HCl when the color salt contained it. The dyes crystallized well, the readiness of crystn. decreasing as the mol. wt. of the dyes rose. The solubility of the tannin compds. increased with the mol. wt. of the resp. dyes. Contrary to the records, weak colloidal properties were not established in the pure dyes, the Brownian movements being seldom noticed; the tannin was of course colloidal and this characteristic rendered difficult the complete removal of excess of dyestuff from the pptd. compds. The reating proportion was established by titration of the tannin with the dye soln. until filtration showed

excess of dye. The results indicate that I mol. of dyes combines with one of tannic acid. Some authorities explain the retention of tannin in the ppt. in amt. above the equimol. proportion here established, by claiming polybasic character for the dye stuffs but S. attributes such results to mere absorption, it being impossible to remove by washing excess of tannin from the ppt., but when the dye is in excess of the tannin it is easily removed by washing, another demonstration of its lack of colloidal character.

[JOSEPH HAROLD.

The Use of "Chromate "Colors for Hat Dyeing. V. FLICK. Farber Ztg., 22, 245-6.

A. S. HALLAND,

Oxamine Derivatives of Anthraquinone which have the Property of Dyeing with Metallic Mordants. E. NOBLTING. Mülhausen. Chem. Zig., 34, 940.—By introducing 2 OH groups, or 1 OH and 1 CO group, or 1 OH and 1 NO or what is equiv., an oxyquinone and an oxime, or finally 2 oximes in ortho position in a chromogen group, well characterized mordanting dyes are obtained. In the anthraquinone series at least the OH and amino group in the ortho position give good mordanting dyes.

E. J. WITZEMAN.

Aniline Black from Aniline and p-Phenylenediamine. Jos. Langer and Th. Kunkel. Farber Ztg., 22, 225-6.—For printing goods which do no stand harsh treatment the writers recommend the use of a printing paste containing 20 g. p-phenylenediamine as formate with only 60 g. aniline salt for each kg. paste. A deep black shade is obtained and no weakening of the fiber observed.

A. S. HALLAND.

Hyposulfites Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>. Aug. Peters. Leipziger-Färber Ztg., 60, 229–32.—Reviews the numerous patent methods for making hydrosulfites. A. S. Halland.

The Fixation of Indigo on Cotton. A. Binz. Farber Zig., 22, 205-6.—It is generally supposed that indigo when dyed on cotton in the vat merely adheres mechanically to the fiber. If this be true, indigo in powder form and on the fiber should show the same reactions. This actually holds good with several reagents heretofore tried, e. g., HNO<sub>2</sub>, AcOH-H<sub>2</sub>SO<sub>4</sub>, NH<sub>2</sub>OH-NaOH, etc. The writer has observed a distinctly different behavior towards C<sub>2</sub>H<sub>2</sub>ONa. All indigoid colors react with this substance as:

$$C_{e}H_{e} \stackrel{CO}{\swarrow} C = C \stackrel{CO}{\swarrow} C_{e}H_{e} + C_{2}H_{6}ONa = C_{e}H_{e} \stackrel{CONa}{\swarrow} C - C \stackrel{OC_{2}H_{6}}{\swarrow} C_{e}H_{e}$$

The reaction is accompanied by a change in color from blue to green and takes place in a few min. Indigo on cotton, linen or artificial silk does not react at all with C<sub>2</sub>H<sub>4</sub>ONa, unless the goods have been steamed for a long time after the dyeing. It must therefore be assumed that indigo combines with the vegetable fiber and that this compd. is loosened by steaming. Indigo printed according to Elbers "indigo gray process" with thickening and oil reacts with the C<sub>2</sub>H<sub>4</sub>ONa, and so does indigo dyed from a neutral alc. soln. of indigo white.

A. S. HALLAND.

Electrolytic Bleaching Liquors. S. H. Higgins. Papier-jabrikant, 9, 564-8; J. Soc. Chem. Ind., 30, 185-8.—A comparison of the relative stability and bleaching effects of (1) bleach liquor made by electrolysis of NaCl soln. (2) bleaching powder soln., and (3) NaClO soln. While possessing some advantages the electrolytic bleach liquor may be used with profit only in localities where salt and power are cheap. Bleaching powder soln. costs \$75 per ton of available Cl, while electrolytic bleach costs \$60-\$115, depending on locality.

V. Nunez.

Action of Carbon Dioxide in the Bleaching Process. S. H. Higgins. Proc. Chem. Soc., 27, 67; J. Chem. Soc., 99, 858-66.—The addition of CaCl, to lime water increase

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the rate of attraction of CO<sub>2</sub> from the air. An exposed soln. of bleaching powder (Ca salts and Ca(OH)<sub>2</sub>) behaves in a similar way. The Ca(OH)<sub>2</sub> gives rise to a deposit of CaCO<sub>2</sub> and then the CO<sub>2</sub> acts on the Ca(OCl)<sub>2</sub>, increasing the bleaching efficiency of the soln. The bleaching efficiency of NaOCl soln. is increased by adding NaCl or other neutral Na salts. Equiv. proportions of NaCl and CaCl<sub>2</sub> have approx. the same effect in increasing the activity of a soln. of bleaching powder. These effects are probably due to the so-called "neutral salt action" and to the increased attraction of CO<sub>2</sub>. The reactions are expressed as follows: Ca(OCl)<sub>3</sub> + CO<sub>3</sub> + H<sub>2</sub>O = CaCO<sub>3</sub> + 2HOCl; CaCl<sub>2</sub> + 2HOCl + CO<sub>3</sub> = CaCO<sub>3</sub> + H<sub>2</sub>O + 2Cl<sub>2</sub>. The bleaching is done by both HOCl and Cl<sub>2</sub>. Cf. von Tiesenholt, J. prakt. Chem., [2] 63, 30; 65, 512; 73, 301; Taylor, C. A., 5, 1238; Higgins, (preceding abstr.).

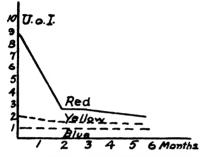


fig. shows the curves for fuchsine.

A Uniform Method for Testing Light Fastness. RALPH L. VON KLEMPERER. Faster Zig., 22, 209-11.—The use of Kallab's color comparator is suggested (see C. A., 2, 473). The dyed material is, previous to exposure, examined in a Kallab app. and its shade detd. in units of intensity of the primary colors, red, yellow and blue. After exposure the dyeing is re-examined. It is convenient to plot the results in a system of coordinates, the abscissa being units of time and the ordinates units of intensity. The

Bleaching Liquors (HIGGINS). 23.

Dyeing, Tanning and Vulcanization Processes (DREAPER). 26.

Viscose (Ost, et al.). 10.

MITCHELL, C. A. AND PRIDEAUX, R. M.: Fibers used in Textil Fabrics and their Application. New York: Van Nostrand. 279 pp., \$3.00.

RISTENPART: Chemische Technologie der organischen Farbstoffe. Leipzig: J. A. Barth. 9 M.

Sansone: Kompendium der Färberei-Chemie. Wien: A. Hartleben. 10 M.

TROTMAN, S. R.: The Principles of Bleaching and Finishing Cotton. London: Chas. Griffin & Co. 8°, 341 pp., \$4.00.

Brit., 20,338, Aug. 31, 1910. BAD. ANILIN- & SODA-FABR., Ludwigshafen a/R, Ger. Mfg. dyes of the anthracene series by treating o-halogen-benzoyl-2-aminoanthraquinone or a deriv. thereof with a substance having an alk. reaction

Brit., 30,284, Dec. 30, 1910. FARBEN. VORM. F. BAYER & Co., Elberfeld, Ger. Mfg. dyestuffs by converting urea or thiourea compds. by treatment with COCl<sub>2</sub>, CSCl<sub>2</sub>, or their equivs., 2 mols. of a p-aminobenzeneazo-1,8-aminonaphtholsulfonic acid (produced by combination of p-nitrodiazobenzene with 1-amino-8-naphtholsulfonic acid in acid soln. and by reduction of the compds. thus produced or by combination of diazotized acidyl-p-phenylenediamines with the said acids and subsequent sapon. of the compds. thus obtained) or of homologs or substitution products thereof substituted in the benzene nucleus, or a mixt. of 1 mol. of these compds. with 1 mol. of a p-aminobenzeneazoarylsulfonic or carboxylic acid or of homologs or substitution products thereof.

Ger., 230,400, July 23, 1909. Addition to 230,399, Mar. 6, 1909; C. A., 5, 2733. BAD. ANILIN- & SODA-FABR. Fixing on vegetable fibers, by employment as vat dyes, the condensation products obtained according to 222,205 and 222,206 by the condensation of aminoanthraquinones with halogen diketones of the type H1.R.CO.CO.R.HI or with the halogen derivs. of phenanthraquinone.

Ger., 235,364, Apr. 26, 1910. Cassella & Co., Frankfurt a/M. Mfg. blue sulfur containing cotton dyes by heating with alkali polysulfides, halogen-contg. indophenols, or leucoindophenols, which are obtained by the condensation of p-nitrosophenols with halogen derivs. of carbazole and N-alkylcarbazole or by the condensation of halogen derivs. of p-nitrosophenol with carbazole, N-alkylcarbazole or its halogen substitution products. Properties are specified.

Ger., 235,631, Nov. 16, 1909. Addition to 234,961, Aug. 6, 1909; cf. C. A., 5, 3168, 2967, 2969. BAD. ANILIN- & SODA-FABR., Ludwigshafen a/R. Manuf. of halogen derivatives of indigo. Penta- and hexahalogen derivs., as well as tri- and tetrahalogen derivs., may be produced from 4,4-dihalogen indigo, 4,4'-dihalogen indigo white, and 4,4'-dihalogendihydro indigo. Especially the pentahalogen compds. possess a better sol. and purer greenish shade. Also the shade of the hexahalogen indigo produced in this way is considerably greener than that of the highly halogenated indigo derivs. obtained by the direct halogenization of indigo or of other halogen indigoes. As initial material either 4,4-dihalogen indigo, 4,4'-dihalogen indigo white, or 4,4'-dihalogendehydro indigo, the salts of bisulfite compds., can be employed, also the halogenization can be effected in different steps. Further details are specified.

Ger., 235,776, Sept. 14, 1909. R. Wedekind & Co., Uerdingen, Niederrhein. Mfg. acid dyes of the anthracene series by condensing the sulfonitro derivs. (obtained by the sulfonation of anthraflavic acid with or without the addition of Hg and H<sub>2</sub>BO<sub>3</sub> and subsequent nitration) such as dinitrodisulfoanthraflavic acid (cf. 99,874, Chem. Centr., 99, (1) 464) with aromatic amines with or without the addition of condensation agents. The condensation is preferably effected in the presence of the HCl salt of the corresponding aromatic amine. Further details are specified.

Ger., 235,775, July 5, 1910. Addition to 230,594, Dec. 31, 1909; cf. C. A., 5, 2735. FARBEN. VORM. F. BAYER & Co., Elberfeld. In the manuf. of monoazo dyestuffs, instead of naphtholsulfonic acids specified in the principal patent, acidylized aminonaphtholsulfonic acids, such as 2-acetylamino-5-naphthol-7-sulfonic acid, are combined with the diazo compds. of aminoarylsulfonamides, such as 2-toluidine-4-sulfo-o-anisidine.

Ger., 235,811, Jan. 25, 1910. FARBW VORM. M. L. & B., Höchst a/R. Mfg. a green-brown vat dye by condensing  $\beta$ -naphthindoxyl with acenaphthenequinone. The condensation can be effected in HOAc, neutral, or alk soln. or suspension, and the acenaphthenequinone can be replaced by its reduction product (C. A., 4, 3304) in which case the leuco compds of the new product is obtained. Properties are specified.

Ger., 235,844, Apr. 3, 1910. T. INOUYE, Shizuoka and T. Mochida, Tokio, Japan; cf. C. A., 5, 3162. In the degumming of raw silk, the outer fibers unwound from the cocoon, and yielding the floss-silk, are taken moist, and cleaned and dried in the known manner in the moist state.

Ger., 235.948, May 16, 1909. CHEM. FABRIK GRIESHEIM-ELEKTRON. Yellow disazo dyestuffs; see C. A., 5, 2432.

Ger., 236,242, June 8, 1909. S. COURTAULD & Co., London. App. for the production of artificial silk and similar threads with a hollow spindle driven between its

bearings and carrying the spinning nozzle. The end of the spindle carrying the nozzle is mounted in a ball socket and the rear end of the spindle is supported by an axially bored stationary pin to which the spinning mass is led through radial openings.

Ger., 236,767, Dec. 3, 1910. FABR. DE PRODUITS CHIM. DE THANN & DE MUL-HOUSE, Thann, Elsass. Printing of hematin. If the readily sol. alkali salt of hematin is dissolved in a thickening agent and then acidified, the hematin is obtained in a colloidal form which prints well when mixed with the mordant and upon steaming is quickly and more completely fixed than the heretofore employed hematinhis. If the compd.

# 26. PIGMENTS, RESINS, VARNISHES AND INDIA RUBBER.

A. H. SABIN, THEODORE WHITTELSEY.

Adaptability of Ores to Manufacture of Pigments. E. W. Buskerr. Mining World, 35, 57-8.—Ores must contain the equiv. of 30% PbO + ZnO. Detailed description of plant, with cost and operation, is given.

A. H. Sabin.

Action of Light on Pigments. A. EIBNER. Chem. Ztg., 35, 753-5, 774-6.— Tests were made by painting panels which were then treated as follows: 1/2 covered with window glass, 1/2 not covered; or 1/2 covered by opaque cover, 1/2 by glass, 1/3 not covered. Pigments were tested alone, also in admixt. with ZnO, and in some cases with BaSO4, CaCO2, white lead, and lithopone. In general, these white pigments had no effect, except MnO, which in almost all cases induced or accelerated light-action, either bleaching or darkening, more marked under glass; with 1 or 2 exceptions, light-action was much greater under glass than when exposed without glass. Tests were made of the most permanent coal-tar colors, also HgO, HgI, HgS, PbO2, PbO, Pb2O2, Co, Cd, and chrome yellows, etc. ZnO added to water colors (pulp colors) "very greatly accelerated" changes when exposed in films.

A. H. Sabin.

A New Accelerated Test for Paints. H. K. Benson and C. Pollock. J. Ind. Eng. Chem., 3, 670.—Paint for steel is applied, two coats, to steel electrodes and its insulating efficiency is taken as inversely proportional to the wattage at the end of 6 hrs., current 8 volts, D. C.

A. H. Sabin.

Fish Oil as a Paint Vehicle. M. Toch. J. Ind. Eng. Chem., 3, 627.—Winterbleached menhaden oil may be mixed 3 parts to 1 part linseed oil for exterior use; used with dryers; superior for some purposes to linseed oil. Whale oil, etc., not satisfactory.

A. H. Sabin.

Quantitative Estimation of Colophony in Residues of Coal-tar Distillation. Holder AND MEISTER. Chem. Zig., 35, 793.—Digest with reflux condenser 2.5 to 5 g. sample with 200 cc. Et<sub>2</sub>O, filter, wash the residue with Et<sub>2</sub>O 3 times, shake the Et<sub>2</sub>O soln. with 0.1 N NaOH (aq.) until the H<sub>2</sub>O soln. is colorless, agitate the NaOH sol. with 50 cc. Et<sub>2</sub>O twice; shake the combined Et<sub>2</sub>O exts. with 30 cc. 0.1 N NaOH, decompose the total NaOH soln. with excess of dil. H<sub>2</sub>SO<sub>4</sub> in the presence of Et<sub>2</sub>O in a separatory funnel, wash the Et<sub>2</sub>O sol. free from H<sub>2</sub>SO<sub>4</sub>, filter, evap. to 100 cc, add 0.5 g. dry boneblack, b. 10 min. on a water bath to remove coloring matter (does not absorb rosin), filter, wash the boneblack with Et<sub>2</sub>O, evap. to dryness, dry 5 min. at 105°, cool and weigh. Add 8% as a correction for the portion of the rosin insol. in 0.1 N NaOH. Greater accuracy than by the Gladding or Twitchell methods is claimed.

E. J. SHEPPARD.

Cremona Varnish. A. LIVACHE. Bull. soc. encour. ind. nat., 125, 9.—Review

of a paper, not otherwise published, of L. Greilsamer; contains a complete review of previous work on early violin varnishes, but no conclusions as to formula.

A. H. SABIN.

Examination of Linseed Oil. C. NIEGEMANN. Chem. Ztg., 35, 893.—Replies to H. Wolff (Chem. Ztg., 35, 350), that in an earlier paper N. referred to heavy mineral oil used as an adulterant for linseed oil, whereas Wolff understood it to be benzine used to thin heavy boiled oil.

A. H. Sabin.

Fatty Coniferous Oils. C. GRIMME. Chem. Ztg., 35, 925-6.—The following oils have good drying properties and are of importance in the lacquer and varnish industry. Pinus silvestris L., Pinus montana Mill., P. cembra L., P. picea L., P. abies L., P. gerardiana Wall, P. pinea L., Cuperssus sempervirens L. (var. horizontalis) Mill., Thuja occidentalis L. The oil content of the seeds varies from 10.8-35.7%. The constants vary as follows: D<sub>10</sub> 0.9298-0.9326; n at 35-40° 1.4608-1.4879; f. p. —4 to —29°; free fatty acids as oleic acid 0.18-2.75%; sapon. no. 186.7-192.0; ester no. 184.3-191.1; I no. 118.3-156.3; fatty acids 89.75-92.26%; glycerol 10.25-10.44%; unsapon. 0.92-3.43%. The constants of the fatty acids vary as follows: m. p. 0 to —16°; f. p. 1 to —19°; n at 35-40° 1.4607-1.4895; neutralization no. 185.7-196.7; I no. 120.8-158.0; mean mol. wt. 285.2-302.1.

E. Scherubel.

The Analysis of Shellac. A. C. LANGMUIR AND F. S. WHITE. J. Soc. Chem. Ind., 30, 786-9.—A method for detg. rosin in shellac (J. Soc. Chem. Ind., 24, 12 (1905)) was recommended by a sub-committee of the Am. Chem. Soc. in 1907 (C. A., 3, 2748) as an official method and has since been the basis on which shellac has been bought and sold in the U.S. Experience has shown that differences between analysts may be traced to a neglect of the conditions controlling the results, i. e., temp., time, conc. and strength of acid used. The Hanus and Hubl solns. offer no advantages over the Wijs. The Endemann method for rosin (C. A., 2, 3060) is shown to be quite unreliable. Shellac wax is detd. by dissolving 100 g. of shellac in 500 cc. H<sub>2</sub>O containing 20 g. Na<sub>2</sub>CO<sub>2</sub>. The wax in suspension is decanted from the sandy residue and collected in weighable form by b. with a known wt. of paraffin (about 15 g.). Orpiment may be present up to 0.15% in crude shellac and to a lesser extent in varnish and bleached shellac. The latter is examined for As by moderately heating 10 g. with 50 cc. HNO, in a 300 cc. Kjeldahl flask until soln. is effected. B. a few mins., add 15 cc. H<sub>2</sub>SO<sub>4</sub>, b. to fumes, add 50 cc. of a satd. soln. of SO<sub>2</sub>, evap. dry, add 60 cc. H<sub>2</sub>O, b. to 40 cc., filter, wash until filtrate amounts to 75 cc. and test an aliquot part by Sanger's quant. Gutzeit test (C. A., 2, 975). The presence of special denatured alc. in grain alc. shellac varnish is most readily detd. by Deniges' acetone reaction with HgSO4; 2% of the denatured alc. is readily detected. Refined Me alc. is detected by Vorisek's method (C. A., 3, 2922) an admixt. of 2% may be shown. The common adulterants of shellac varnish are rosin, spirit-sol. copals and sandarac. Rosin is approx. detd. by comparative colorimetric tests with Wijs soln. Copal and sandarac give a less pronounced red color than rosin and a curdy ppt. which may also be produced by diln. of the varnish with alc. or glacial AcOH. A. C. LANGMUIR.

The Chemistry of Caoutchouc. Theory of Vulcanization. D. Spence and J. H. Scott. Z. Chem. Ind. Kolloids, 8, 304.—Ostwald's conclusions according to which the combined S of vulcanization is physico-chemically combined or adsorbed by the hydrocarbon do not fully explain the facts known in regard to vulcanization. Regenerated but not devulcanized rubber requires and combines with the same amt. of S on revulcanization as it required originally and in the bromination of soft cured rubber a stoichiometric relation between S and Br undoubtedly exists  $(S_2 = Br_4)$ . The authors have worked in the so-called free S field instead of in the combined S field as did Ost-

wald and Buizov. By repeated quant. extraction of standard samples of vulcanized rubber by known and equal vols. of b. acetone in an app. designed for this purpose the authors have shown that the extraction process follows the curve of a typical adsorption isotherm. The value of 1/n in the adsorption equation  $C = (a_2^n/\frac{1}{n})$  is calc. from their results and is found to be in good agreement with the value found for other adsorption processes. The authors conclude that: (I) The S of vulcanization is chemically combined with the caoutchouc and is not merely adsorbed (II); free S only is adsorbed; (III) the so-called free S of vulcanization really consists of adsorbed S and a certain amt. of free S depending upon the conditions of vulcanization. The adsorption of free S by the disperse phase of the rubber is considered as a stage in the subsequent chem. reaction between the rubber and S. Complete extraction of the S of vulcanization cannot be effected with acetone or with alkali. Curves in illustration of the extraction process are given. [For corrections to this paper see *Ibid.*, 9, 48.—Abstr.].

Suggestions for Standard Testing of Rubber, by International Rubber Testing Committee. Gummi Ztg., 25, 1277.—Breuil suggests testing of strips instead of rings, criticizes requirements of Schoppen-Dalen machine and thinks any dynamometer with error less than 1% good enough for elongation and hysteresis curves. He values tests under conditions of use in the field. He thinks 20 min. washing too long, as it may affect the viscosity. He recommends a trial of viscosity determined with other apparatus such as a tube or falling ball in rubber solution; does not understand why such complex procedure is necessary for valuation of raw rubber; it should be tested as it is manufactured, for tension, pressure, friction, etc. Frank replies that in order to obtain conclusions from valuable results, they should be comparable and they will only be so if all use the same apparatus and procedure. Experience shows that the ring form of test piece is better than the strip form and also that 20 min. washing is without effect upon the viscosity. Viscosity determination is important in the technical valuation of rubber in the opinion of experienced rubber chemists.

M. H. DANIELS.

Valuation of Rubber. F. Frank. Gummi Ztg., 25, 1482.—Frank and Breuil repeat the above arguments (preceding abstr.).

M. H. D.

New Method of Coagulation. Anon. Gummi Ztg., 25, 365.—In Dern's method for treating Mexican latex the milk is tapped and filtered the same day, coagulating powder is added and then allowed to ferment, after which it is preserved or shipped in air-tight lead cases. In the factory it is treated with a second compound made by Dern, 2 liters to 5 gals. latex. The mixture is centrifuged 30 min., sepg. resins and proteins. Yield from Castilloa, 35% pure rubber, 10% resin rubber; from Hevea, up to 40% pure rubber, 6% resin rubber. Value of resin rubber covers cost of production. Pure rubber washed and dried and shipped in 24 hrs. In a plant of 2 presses, 20 machines can handle 200 gals. latex per day. Coagulating compound may be distilled and used again by strengthening with 20% alc. It is apparently alcohol and acetone.

M. H. Daniels.

New Views on Vulcanization of Caoutchouc. E. STERN. Gummi Ztg., 25, 836, 926.—Vulcanization is considered to consist of a mechanical solution of S in rubber, followed by the addition of  $S_z$ . It is experimentally possible to start with melted S and by dissolving increasing amounts of rubber in it to produce alloys. If S forms a compound with rubber  $C_{10}H_{16}S_3$  (32% S — Weber) then the multiples of the rubber-S alloys will first fall to an eutectic, then rize to 68%  $C_{10}H_{16}$ , 32% S and then fall again. Study of the reaction in a homogeneous system such as melted naphthalene is suggested to test the equation: (dx/dt) = k.  $C_{rubber}$   $C_{sulfur}$  where k = speed const. But the

course of vulcanization never takes place in a homogeneous system. A theory of vulcanization will have to take into consideration the phys.-chem. idea of surface reaction and he gives a brief review of adsorption, especially as applied to cold vulcanization where the progress is detd. by speed of diffusion of S<sub>2</sub>Cl<sub>2</sub>. S. coincides with Hinrichsen's view that the adsorption hypothesis is not far reaching enough and that vulcanization is a result of both adsorption and purely chemical change. M. H. D.

Determination of Caoutchouc as Tetrabromide; Decomposition of the Bromide by Nitric Acid. D. S. Spence, J. C. Galletly and J. H. Scott. Gummi Markt, 5, 113; see C. A., 5, 3172.

T. W.

Determination of Caoutchouc by Budde's Tetrabromide Method: Influence of Protein. D. Spence and J. C. Galletly. Gummi Markt, 5, 109; cf. following abstr.—Budde's method in authors' hands has given results varying in extreme cases by as much as 10% from true value; due to conditions not yet fully understood, but apparently connected with the formation of the tetrabromide and its subsequent decomposition. A carefully planned series of analyses shows that protein bodies and inorganic constituents are precipitated with the tetrabromide, rendering the gravimetric determination inaccurate. As the Br absorption by such substances is slight the volumetric method is not seriously affected.

C. E. Bradley.

Determination of Caoutchouc in Crude Rubber. Jules Bock. Rev. gen. chim., 14, 209-21.—A comparison of the Budde method as modified by Fender and Kuhn (gravimetric) (C. A., 2, 593) and the recent volumetric method of Budde (C. A., 3, 3013) applied to 12 samples of crude rubber. Results with the grav. method were higher, due to contamination of the tetrabromide with rubber protein. Washing and length of Br treatment also influences results. Resins do not.

C. E. Bradley.

A Comparison of the Dyeing, Tanning and the Vulcanization Processes. W. F. DREAPER. Z. Chem. Ind. Kolloide, 8, 253-60; cf. C. A., 5, 1843.—A theoretical and comparative study, dealing with the many points of similarity in the 3 processes mentioned. In the initial fixation of the dyestuff by the fabric, of the tannin by the hide and of the S of vulcanization by the rubber D. believes that analogous forces are at work. These forces are of physical origin and bring about zones of increased conc. (leading to pptn. in some instances) around the surface of the particles forming the disperse phase (adsorption phenomena). Surface tension may be the driving force or the neutralization of elec. charges of opposit sign may be responsible for the results produced as in the case of the pptn. of the dyestuff on the fabric (theory of Perrin). No one physico-chem. theory serves to explain every observation made in regard to the processes in question. In the final stages of the dyeing and of the tanning process chem. and physical forces probably intervene. The modern "adsorption theory" of vulcanization is touched upon.

Rapid Determination of Fillers in Rubber Compounds. BRUGGEMAN. Gummi Ztg., 25, 1529.—By the method of the Imperial Testing Labs., a portion of the compd. is dissolved in b. petroleum, the resulting soln. dild. with benzine and filtered through a Gooch, or centrifuged. Turbid filtrates often result. If filtration is effected in a Gooch, fitted with double filter plates, which has been filled half full with kieselguhr, clear filtrates, are obtained, and the length of time required for filtration is shorter. The same crucible may be used again, provided a layer of the kieselguhr (about 2 mm.) is removed from the upper surface.

R. T. Stokes.

Kickxia Rubber. II. F. Frank and E. Marchwald. Gummi Ztg., 25, 877.—Systematic testing out of various coagulating agents, and comparative physical and chem. tests on the rubber obtained. Well aged latex gives better quality of rubber

than fresh latex. Energetic coagulating agents must be used to obtain products of high resiliency. A plantation Kickxia rubber equal to first class Para or Congo rubber can be produced.

R. T. STOKES.

Prevention of "Tackiness" in Raw Rubber. E. FICKENDEY. Z. Chem. Ind. Kolloide, 9, 81-3.—General observations m regard to the behavior of rubber in an indifferent atm. and in air and O, lead the author to believe that "tackiness" is essentially the result of oxidation. He recommends therefore the addition of such substances (particularly colloid substances) to india rubber as will combine with and take up O readily, for example an appropriate amt. of a dil. soln. of tannin. Rubber so prepared darkens in color but does not become "tacky." [This may be perfectly true but can hardly be taken as showing that tackiness is the result of oxidation. We know furthermore to-day several measures by means of which "tackiness" can be effectively prevented. It still remains to be shown by more elaborate and extensive tests of what value the new suggestion is.—Abstr.]

CHRISTY, C.: The African Rubber Industry and Funtumia elastica. London: Bale. 8°, 252 pp., 12 s. 6 d.

Brit., 7,722, Mar. 30, 1910. R. C. CHILD, 11 Queen Victoria St., London, E. C. A printing ink consisting of the mixt. of a tarry body having a considerable % of pitch, with powdered shale or anthracite coal.

Ger., 235,594, Nov. 24, 1908. ALEXANDER and POSNANSKY, Copenick bei Berlin. In the manuf. of objects from factis, the action of S or S chloride on fatty oils is interrupted by cooling as soon as the mass is converted into a plastic or viscous product, the objects are formed from the mass alone or after mixt. with other substances, and then the factis formation is effected at a high temp.

Ger., 235,757, Sept. 11, 1909. J. MEURANT. Paints and coating masses; see C. A., 5, 2979.

Ger., 236,034, Dec. 12, 1909. FARBW. VORM. M. L. & B. Yellow-green pigment dyes; see C. A., 5, 3175.

Ger., 236,338, Apr. 9, 1909. S. WECHSLER, Vienna. In the manuf. of printing colors for printing rolls etched in relief, the dry powdered color to be used, without previous soln., is intimately mixed with varnish and glycerol, and to the resulting mass a mixt., of the known kind, of glycerol, an alkali salt-such as soda or argol, and resin oil. and varnish (hygrol), is added.

# 27. FATS, FATTY OILS AND SOAPS.

#### E. SCHERUBEL.

Enzymes and their Application in the Fat Industry. F. VIGNOLOLUTATI. Ind. chim., 11, 200-2.—Brief review, based chiefly on Welker's work (C. A., 5, 2112) on the actio 1 of the enzyme of the castor bean.

CHAS. A. ROUILLER.

Lubricants. I. H. Schlütter. Papier Fabrikant, 9, 1036-40.—Well-known methods of detg. physical constants of lubricating oils. II. Ibid., 1061-6.—Chem. methods of testing lubricating oils and classification.

E. Schlützer.

Heatsfoot Oil. W. Fahrion. Seifensieder Ztg., 38, 925-6.—Discussion of adulterations E. S.

Motor Cylinder Oils. C. F. Otto. Seisensieder Ztg., 38, 629-30, 657, 680-1.

Examination of Copra. W. HÜPFNER. Chem. Ztg., 35, 872.—The author's method accords with that of the association of German oil mills.

E. S.

Soy-bean Oil. S. KEIMATZU. Chem. Ztg., 35, 839-40.—From his investigations, the author has detd., (1) that the oil contains 0.2% pytosterol in which stigmasterol cannot be detected; (2) there are 12% of satd. fatty acids mainly stearic and palmitic; (3) there are about 80% of unsatd. fatty acids of which about 50% consist of an isomer of linoleic acid which yields an hydroxyacid of m. p. 158-9°. Besides these oils there is present linoleic and oleic acid.

E. SCHERUBEL.

Machine Oils. C. F. Otto. Seifensieder Ztg., 38, 539-40, 582.—Recipes. E. S.

Determination of Sesame Oil. H. Kreis. Basel. Chem. Zig., 34, 1040.—The problem was to det. whether a peanut oil contained several % or only traces of sesame oil. The sample gave all typical color reactions for sesame oil with furfural-HCl, ZnCl<sub>2</sub>-HCl, HNO<sub>2</sub>, resorcinol-HNO<sub>3</sub> and H<sub>2</sub>O<sub>3</sub>-H<sub>2</sub>SO<sub>4</sub>. The latter reaction does not appear definitly with less than 5% sesame oil. The sesamine test was found to be best for detecting traces. By using 150 cc. of oil containing 1-5% sesame oil sesamine was detected microscopically by the Bömer's color reaction with Ac<sub>2</sub>O and conc. H<sub>2</sub>SO<sub>4</sub>

E. J. WITZEMANN

A List of Waxes. II. D. J. LEMAL. Metal. Ind., 9, 372; cf. C. A., 5, 3351.
J. J. M.

Fatty Coniferous Oils (GRIMME). 26.
Products of the Soy Bean (BELTZER). 12.

HURST, G. H.: Lubricating Oils, Fats and Greases. Their Origin, Preparation, Properties, Uses and Analysis. 3rd Rev. Ed. New York: Van Nostrand. 400 pp., \$4.00.

LEIMDÖRFER: Beiträge zur Technologie der Seife auf kolloid-chemischer Grundlage. Dresden: T. Steinkopff. 1.80 M.

RANSOM, L. A.: The Great Cottonseed Industry of the South. New York: Oil, Paint and Drug Reporter. 8°, 128 pp., \$1.25.

STIEPEL: Fette, Oele, Wachse usw. Leipzig: J. A. Barth. 5 M.

Brit., 7,726, Mar. 30, 1910. N. TESTRUP, 6 Broad St. Place, London, E. C. Treating oils, fats, and the like, by exposing them while mixed with finely divided catalyst as a spray or in a similar condition of minute subdivision in an atm. of H or free H-contg. gas.

Ger., 236,488, Aug. 6, 1910. VER. CHEM. WERKE, Charlottenburg. In the manuf. of saturated fatty acids and their glycerides from the corresponding unsatd. compds. by the action of H, Pd or palladous hydroxide, pptd. from indifferent substances, is used as the H carrier. Details and modifications are specified.

# 28. SUGAR, STARCH AND GUMS.

A. HUGH BRYAN.

Modern Prospects in the Sugar Industry. F. Mansfield. Z. Zuckerind. Böhmen, 35, 651-2.—A discussion of the prospects of the further application of electricity in

the sugar industry, for power, heating, and possible sepn. of sugar by electrolytic means.

WM. E. Cross.

Density of Liquid Sugar and of its Aqueous Solutions. F. Schwers. London. Davy Faraday Res. Lab. Bull. sci. acad. roy. belg., 1911, 503-16; J. Chem. Soc., 99, 1478-85.—The d. of superfused sugar was detd. by m. in a pear-shaped pycnometer under paraffin (b. about 190°). All wts. were reduced to a vacuum. The values found for d., referred to H<sub>2</sub>O at 4°, were as follows: —14.55°, 1.51966; 0.00°, 1.51915; 14.00°, 1.51756; 20.55°, 1.51663; 33.75°, 1.51245; 45.85°, 1.50605; 59.00°, 1.49812; 77.85°, 1.48493; 101.55°, 1.46790; 114.70°, 1.45846. The slope of the curve indicates the existence of a max. d. but this is not yet reached at —15°. Using these values for the real d. of sugar in soln. and Gerlach's for that of aq. solns. and thus detg. the contraction resulting when sugar is dissolved, it is found that the contraction decreases up to about 50°, then increases to a max. at about 80°, and again decreases; i. e., the phenomena are analogous to those observed in the binary systems H<sub>2</sub>O-alcohols (EtOH, glycol, glycerol; cf. C. A., 5, 14, 613, 614, 814). Chas. A. ROUILLER.

The Solubility of Lime in Sugar Solutions. H. CLAASEN. Z. Ver. Zuckerind., 61, 489-509.—The soln. of the lime was made in a special app., by which the temp. could be well controlled, and evapn. prevented. The amt. of lime dissolved after a certain time was obtained by titration of a measurued wt. thereof against standard acid. The solubility of quicklime from different sources was the same, while lime added as quicklime was more sol. than that added as milk of lime. The presence of excess of added lime slightly increased the solubility, up to a certain point only (2-2 1/2%). The solubility decreased with increase in temp., but the rate of soln. increased; it also increased with the conc. of sucrose in the soln.; not only in relation to the wt. of soln. but also to the wt. of sugar. Sugar solns. which are satd. with lime at low temps. (in presence of excess lime) deposit lime, but no saccharate, when heated. The cold filtered satd. solns. on heating, however, deposit lime and saccharate—the latter as a gelatinous mass. A soln. made at 50° deposits no saccharate on heating to 90°. It is concluded that in sugar manuf, the lime can cause no pptn, of sugar either on heating or cooling the soln. WM. E. CROSS.

The Behavior of Sucrose and its Decomposition Products on Warming. I. E. DUSCHKY. Z. Ver. Zuckerind., 61, 581-608.—The effect of heat on sucrose, dextrose and levulose was studied. Solns. of pure dextrose showed decrease in polarization on heating (120-140°) in the autoclave, the decomp. increasing with temp. and time. Lactic and acetic acids retarded the decomp.; alkalies and Na salts of weak acids accelerated it. Slight increases in polarization occurred in very conc. solns. and in presence of acids on heating, and this is ascribed to the formation of highly active decomp. products. Further heating always resulted in decreased polarization and slight decrease in reducing power. The decrease in polarization was always small (up to 15%). Levulose was more easily decomposed than dextrose, temps. of 60-100° being sufficient to bring about very considerable decreases in polarization. The amt. of decomp. increases with time of heating, temp. and pressure. Acids retarded, and alkalies accelerated it. Decrease in polarization was accompanied by a loss in reducing power, leading to the view that the decomp. products were optically inactive substances possessing small, if any, reducing power. WM. E. CROSS.

The Occurrence of Levan in Sugars. W. G. TAGGART. J. Ind. Eng. Chem., 3, 646-7.—Sugars on storage deteriorate but the fall in single polarization and in sucrose (Clerget) cannot be accounted for by inversion. In many cases the Clerget sucrose slightly increased. This is believed to be due to the gum levan which on hydrolysis gives levulose.

A. H. BRYAN.

Optically Active Substances in Sugar Manufacture. O. KOPECKY. Z. Zuckerind. Böhmen, 35, 646-51.—A difference between single and Clerget polarization of beet juices is found with almost all beet juices, though it is variable in amt., dependent on climatic and other conditions during growth. The diff. is caused by dextrorotatory non-sugars, not merely raffinose, dextrose and asparagine, which are not pptd. by Pb subacetate, and do not reduce Fehling's soln. Some of these active substances are pptd. during manuf. while others remain in the final products. Some give mucic acid on oxidation. Their unsuspected presence sometimes causes serious unaccountable apparent losses in sugar manuf.; and it moreover discounts seriously the value of chem. control unless on the Clerget basis. These optically active substances were not present in beets until recent years, the beets having now become abnormal, perhaps through degeneration of the plant.

WM. E. Cross.

The Uniform Determination of Dry Substances in Sugar Products of All Kinds. T. KOYDL. Z. Zuckerind. Böhmen, 35, 491-7.—Results on dry substance for raw sugars do not agree with those obtained for syrups because the resp. methods of drying (sugar alone, syrup with sand) are essentially different. In drying raw sugars (heated 1½ hrs. at 110°) the sugar crystals form the foundation for the drying molasses, and it is suggested that sugar crystals should therefore be used instead of sand in drying syrups, and the dry substance of the mixt. obtained exactly as with sugars. In analyses of juices and thin syrups, from which the thick syrups, raw sugars, etc., are to be obtained the results will be comparable if the juices, etc., be dried to syrup first, without sand, and then sugar crystals introduced, and the drying continued thus. The method should be so conducted that the relation of water to sugar is about the same in all cases. As pure sucrose can be heated 14 days without loss in wt.; there is no danger of loss on this ground. The author proposes the name "dry residue" instead of "total solids" or "dry substance."

W. E. C.

The Determination of Gums in Syrup. X. ROQUES AND G. SELLIER. Ann. chim. anal., 16, 218-20.—The authors confirm the claims of Chauvin (C. A., 5, 3353) concerning the value of neutral Pb(AcO)<sub>2</sub> in EtOH soln. as a precipitant for gum. To the wt. of ppt. a correction for amt. of Pb salts present should be applied; this is calc. from the wt. of ash left after careful ignition of the gums, after adding a few drops of fuming HNO<sub>2</sub>. Two methods similar to Chauvin's are given in detail.

H. S. BAILEY.

Notes on the Determination of Acids in Sugar Cane Juice. P. A. YODER. J. Ind. Eng. Chem., 3, 640-6.—The author gives some notes on the analytical operations and some detns. and approx. estns. of acid constituents in cane juice. The acids considered are oxalic, H<sub>2</sub>PO<sub>4</sub>, H<sub>2</sub>SO<sub>4</sub>, tartaric, malic, succinic, aconitic and citric.

A. H. BRYAN.

Manurial Experiments with Sugar Beet. E. SAILLARD. Beidermanns Zentr., 39, 859-60; J. agr. prat., 1910, 1, 267; J. Chem. Soc., 100, II, 145.—Na<sub>2</sub>NO<sub>2</sub> and cyanamide produced about the same amts. of beets and sugar, while Ca(NO<sub>2</sub>)<sub>2</sub> gave higher results. Kainite produced more beets and more sugar than the same amt. of K as Cl. This is due to the Mg present.

A. H. BRYAN.

The Practical Application of Electrolysis to Sugar Manufacture. F. Mansfeld. Z. Zuckerind. Böhmen, 35, 656.—The electrolysis of juices, syrups, etc., would remove the CaO and Fe and would reduce the viscosity.

WM. E. Cross.

Electrolytic Clarification of Beet Juices. F. Mansfeld. Z. Zuckerind. Böhmen, 35, 653-6.—A discussion of the electrolysis of juices as a means of purification. A very strong current would be necessary, and only a partial clarification would result; the method could only be used as an adjunct to present methods. Wm. E. Cross.

Raw Juice Sulfitation. W. H. Th. Harloff. Arch. Swikerind., 19, 819-32.—Raw juice treated with 1% milk of lime was heated to different temps., sulfured, and its color compared with that of the same juice limed and sulfured at 30°. Up to 60°, heating seems to decrease both the color and the CaO content, especially with thin juice; while above 70° both increase rapidly. A juice neutral to litmus settles much faster and filters much clearer than one neutral to phenolph. W. L. BADGER.

A Plea for Single Carbonation. W. H. TH. HARLOFF. Arch. Suikerind., 19, 783-818.—Both single and double carbonations are equiv. as far as color is concerned. The double is longer, more expensive, but neutralization, in lab. tests, is easier (hence, special care is necessary). The acid thin-juice process has its place with both methods and will probably lessen neutralization troubles in the single method. The unsulfured thick juice should be as light-colored as possible. Single carbonation requires less time. The addition of lime some time before satn. has no ill effect on the color, so long as the temp. is not raised, but it increases the ash content; putting alk. juices under pressure, as in filtering, appreciably increases the ash content. The color of the thin juice with both methods is the resultant of the color due to the products of glucose decomp, and the decolorization by removing, with CaO, the original coloring matters in the juice. These latter are easily bleached, the former little or not at all. By careful working, the CaO content may be considerably less with single than with double carbonation. With the double method no more of the Ca salts of glucose decomp. products are removed than with the single. With the single carbonation warming is not permissible, but troublesome acidity of the thin juice is avoided. W. L. B.

Drying in Vacuum. B. VIOLA. Met. Chem. Eng., 9, 389-92; cf. C. A., 5, 3178.—The author describes and illustrates the Passburg vacuum chamber dryers, this being the foundation of the present system of vacuum drying. He also touches upon some other types of app. for the final removal of the last traces of moisture from materials which are desired to be thoroughly and perfectly dry in their final state, such as glue powder, milk or egg powder.

A. H. BRYAN.

Rapidity of the Conversion of Starch to Sugar by Acids. H. Van Laer. Bull. soc. chim. belg., 25, 249-64.—A study is made of the variations of K with time, with starch paste and with sol. starch, with variations in conc. of the starch, with H<sub>2</sub>SO<sub>4</sub>, HCl, oxalic, tartaric and KH<sub>2</sub>PO<sub>4</sub>, and with variations in conc. of the acids. The conclusion is reached that the law for the conversion of starch to sugar by acids and that for the hydrolysis of saccharose by acids are not essentially different whatever the physical condition of the amylaceous material acted upon.

I. K. Phelps.

Action of Oxalic and Malonic Acids on Starch and Dextrin. OECHSNER DE CONINCE AND RAYNAUD. Inst. chim. Montpellier. Bull. sci. acad. roy. belg., 1911, 438-9; cf. C. A., 5, 2443.—Like mineral acids but unlike monobasic org. acids (HCO<sub>2</sub>H and AcOH), oxalic and malonic acids hydrolyze dextrin more rapidly than they do starch, From 1-50 cc. of a satd. soln. of the acids, diluted to 50 cc., reacting 25 min. at 100° on 1 g. of starch and dextrin, resp., hydrolyzed the following % of the carbohydrates (only the values for the extreme conc. of acids are given): Oxalic, 1 cc., 3.65, 6.38; 50 cc., 23.22, 25.62. Malonic, 1 cc., 4.1, 5.7,; 50 cc., 33.7, 41.0. C. A. ROUILLER.

Action of Lactic and Tartaric Acids on Starch and Dextrin. W. OECHSNER DE CONINCE AND A. RAYNAUD. Inst. chim. gén. Montpellier. Bull. acad. sci. roy. belg., 1911, 592-3.—The amts. of starch and dextrin. resp., hydrolyzed (cf. preceding abstr.) are as follows: Lactic, 1 cc., 5.50, 5.60; 5 cc., 27.00, 26.00. Tartaric, 1 cc., 4.7, 5.8; 5 cc., 17.5, 20.0. Chas. A. ROUILLER.

Viscose (OST, et al.). 10.

SPENCER, G. L.: Methods of Analysis and Chemical Control for Use in the Factories of the Cuban-American Sugar Co. New York: The Cuban-American Sugar Co. 8°, 32 pp., \$1.00.

# 29. LEATHER AND GLUE.

#### WALTER J. KIETH.

Some Advantages of the Graphic Method of Recording Results in Routine Analysis. J. H. Russell. J. Am. Leather Chem. Assoc., 6, 382-7.—By plotting the results of analyses of tan liquors extending over a period of time, variations in the comp. of the liquors is readily detected. The method also finds service in showing the trend of tannin costs.

Charles R. Oberfell.

Note on the Calorific Power of Some Tanning Materials. A. W. HOPPENSTEDT. J. Am. Leather Chem. Assoc., 6, 388-9.—The calorific powers of the following tanning exts. in cal. and B. t. u., resp., are: Clarified quebracho 5.265, 9.477; ordinary quebracho 5.710, 10.278; chestnut 4.248, 7.646; hemlock 4.895, 8.811; and oak 4.952, 8.914.

CHARLES R. OBERFELL.

Notes on the Official Methods of the American Leather Chemists Association.

J. H. YOCUM AND T. A. FAUST. J. Am. Leather Chem. Assoc., 6, 420-8.—A criticism of the official methods and suggestions for reform.

CHARLES R. OBERFELL.

The Strength of Acids. H. R. PROCTER. J. Am. Leather Chem. Assoc., 6, 442-5; through Tanners Yearbook, 1911.—HCl and H<sub>2</sub>SO<sub>4</sub>, due to their almost complete dissociation have drastic action on hide substances, while CH<sub>4</sub>.CO<sub>2</sub>H, HC.O<sub>2</sub>H and CH<sub>2</sub>.CHOH.CO<sub>2</sub>H act mildly on hide, because of slight H conc. A strong organic acid can be weakened in its action by adding its own salt, but not in the case of a mineral acid. CH<sub>2</sub>.CO<sub>2</sub>Na added to HCl will reduce its strength to that of HC.O<sub>2</sub>H, so that either HCl or H<sub>2</sub>SO<sub>4</sub> can be made safe for hide. The relative cost of dissolving CaO in various acids is given.

The Neutralization of Chrome Leather. Edmund Stiasny. J. Am. Leather Chem. Assoc., 6, 445-9; through Tanners' Year Book, 1911.—Free acid will cause trouble in fat-liquoring, for the acid will decompose the soap and the free fatty acids may combine with the sol. part of the absorbed Cr salts forming Cr soaps of a sticky and smeary character. Neutralization removes all free acid which cannot be washed out. Free acid also tends to brittle grain. Excess of alkali is equally disastrous. Common practice is to apply litmus paper to a cut section, but this does not insure uniformity. It is not advizable to use a strong alkali; even borax and Na<sub>2</sub>PO<sub>4</sub> require care. A mixt. of equal parts of Na<sub>2</sub>CO<sub>3</sub> and NH<sub>4</sub>Cl gives best results, as the NH<sub>4</sub> is generated as fast as it is used, thereby preventing large excess and insuring complete neutralization.

Chas. R. Oberfell.

Treatment of Tannery Effluents (MORRISON). 14.

Dyeing, Tanning and Vulcanization Processes (DREAPER). 26.

## 30. PATENTS.

Abstracts of patents are included under the foregoing divisions of the Journal. The abstracts of U. S. Patents are prepared by Earl T. Ragan, those of the British, French and German Patents by O. D. Swett and those of Canadian Patents by Russell S. Smart, of Fetherstonhaugh & Co., Ottawa (5 Elgin St.). The abstracts of French

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# CHEMICAL ABSTRACTS

Vol. 5.

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No. 22

### 1. APPARATUS.

L. C. JONES.

A New Cork-boring Machine. F. HANFLAND. Chem. Ztg., 35, 908.

J. H. MOORE.

Simplified Apparatus for Determination of Carbon in Iron. G. BUTZBACH AND G. FENNER. Chem. Ztg., 35, 917.—Three flasks are described. One has the neck narrowed at the top to fit into a rubber stopper in the mouth of the return cooler; in another the flask and cooler connect by a ground joint. In both forms the stream of air enters through a tube passing down the cooler. The third form has a side-tube for admitting the air.

J. H. MOORE.

A New Form of Rubber Stopper. Schweiz Wockschr., 49, 409.—The stopper is shaped in the form of a cap and will fit over the neck of a flask. H. C. FULLER.

The Grasser-Allen Extraction Apparatus. L. ALLEN. Lab. for Oil and Fat-Ind. Chem. Rev. Fett-Harz-Ind., 18, 219 (illust.).—A "sugar funnel" with a stop-cock at its lower contracted stem has inserted into it a glass tube into which the material to be extracted is placed. A reflux condenser is connected at the other end. The solvent is poured in with the stopcock closed, and several extractions are made in the cold; the operation is finished hot as in a Soxhlet app.

P. ESCHER.

A New Extraction Apparatus. G. A. QUINCKE. Z. Nahr. Genussm., 22, 171-2.

—The object is to extract with a warm solvent in a Soxhlet. The whole app. is enclosed in a glass cylinder warmed to the desired temp. No cork or rubber connections are necessary, and efficient condensation is secured. Made by Paul Altmann, Berlin.

D. S. PRATT.

Water Bath with Constant Level, Pre-heating, and Dust Protector. H. Leiser. Chem. Zig., 35, 899.—A reservoir for attaching to ordinary H<sub>2</sub>O baths. When the H<sub>2</sub>O falls to a certain level steam enters the reservoir, pre-heating the H<sub>2</sub>O and allowing it to flow to the bath.

J. H. MOORE.

Automatic Filtration of Liquids at Constant Level. F. C. Bellaire-Wörschweiler. Chem. Zig., 35, 880.

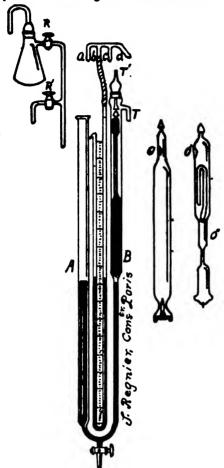
J. H. Moore.

Apparatus for the Simultaneous and Automatic Filling of Several Vessels to Same Level. E. Dumesnil. Bull. sci. pharmacolog., 18, 90-2.—The liquid is siphoned through a several-branched delivery arm from a Woulff's bottle bearing a tube which regulates the siphon level. To the end of each branch of the delivery arm of the siphon is attached a mouth piece consisting essentially of 2 close fitting cylinders each provided with heavy metallic collar and expanded conically below. When ready for use the mouth pieces are suspended, by the collars of the outer tube, in the vessels to be filled. The inner tubes attached to the siphon branches are longer than the outer and are closed at the ends, each being provided with 3 holes on the sides which are carried by the falling inner tube beyond the end of the outer tube when the siphon is started and are closed by the falling of the outer tube when the mouth pieces are being transferred to other vessels.

V. K. Chesnut.

Regulator for Reduced Pressure with Periodic Variations. A. VILLIERS. Bull.

sci. pharmacolog., 18, 7-11; cf. C. A., 5, 806.—This app. permits close control of the pressure of air or gas in a vessel from atm. pressure up to the extent of the vacuus



obtainable. The wt., height and general design of the Hg float in the upper part of B are varied to comply with the nature of the gas or the pressure desired. It has glass points on the sides to control accuracy of motion and is terminated by a glass cone which fits snugly in the conical tube attached to the stopper. If air be in the vessel to be exhausted and it be large, connect T' with the pump, awith R or R', c with T and d with the vessel to be exhausted. The Hg rizes in B until it lifts the float and closes the pump connection with the cone. returning again when the increasing pressure in the vessel permits and then continuing to move up or down with the changes of pressure. If another gas is being handled and a high vacuum be desired in a small vessel, one of the tubes shown to the right of the U-tube is used. Each is pierced by a small hole above. The 1st contains a glass bulb partly filled with Hg and the 2nd a siphon emptying at o''. In this case connect T'with the air or gas, T with c, a with the pump, b with the manometer and d with the vessel to be depleted. On applying the vacuum the ascending Hg raises the bulb wt. thus preventing Hg from entering the float from below and rizes until a sufficient amt. enters o or o' to depress the wt. or set the siphon to playing, when it subsides

as far as the pressure permits and then plays between these limits. V. K. C.

Apparatus for Generating Large Quantities of Hydrogen Sulfide, and Partial Recovery of the Waste Gas from Precipitations. A. Gwiggner. Chem. Ztg., 35, 891.—

The generator delivers to a glass tube with T's for attaching a number of pptg. flasks from which the waste gas is carried through a common tube to a vertical H<sub>2</sub>O scrubber about 70 cm. high, filled with glass beads, the H<sub>2</sub>S-water collecting in a reservoir beneath.

J. H. Moore.

New Partitioned Apparatus for Decomposing Water. K. WOYTACEK. Chem. Ztg., 35, 943.—A graduated tube is divided lengthwise by a partition, each compartment ending at the top in a tube with glass cock. The Pt or C electrodes are placed in the lower end of the tube just below and almost touching the partition. The "level-tube" is attached just below the electrodes and terminates in a bulb above the top of the graduated tube.

J. H. MOORE.

A Practical Laboratory Apparatus for Evolution of Carbon Dioxide. C. PURRMANN AND P. VERBEEK. Chem. Lab., Goldschmieden, H. Bergius & Co. Chem. Zig., 35, 927-8.—A simple arrangement of aspirator bottles for holding the acid and limestone.

J. H. M.

Improvements in Vacuum Distillation Apparatus. E. John. Chem. Ztg., 35, 943.

General Use of the Geryk Pump in Vacuum Distillation. G. Doby. Chem. Ztg., 35, 756.

J. H. Moore.

Apparatus for the Determination of the Specific Gravity and the Molecular Weight of Gases. GULICH. Stendal. J. Gasbel., 54, 699-700.—The app. consists of a large cylinder, a monometer, a cross-bored stopcock in one bore of which is a piece of Pt foil pierced by a fine hole, a thermometer, etc. The cylinder is evacuated, the pressure read on the manometer, air allowed to flow in through the fine hole in the Pt foil, the time necessary for the pressure to rize being noted on the manometer. The cylinder is again evacuated to the same point, the process repeated with the gas of unknown d. and the d. calc. in the usual way.

George W. Morey.

A New Gas Analysis Apparatus. Anon. J. Gasbel., 54, 814-6.—The chief feature of this app. is the special stopcock by which connection is made with 3 different tubes.

George W. Morey.

A New Orsat Apparatus for Technical Gas Analysis. C. HAHN. Aachen. J. Gasbel., 54, 870-1.—Contains little that is new. George W. Morey.

Platinum Electrodes for the Determination of the Concentration of H and OH Ions.

L. v. Liebermann. Chem. Ztg., 35, 972.—The vessels for holding the liquids are improved by fusing on glass feet which fit in cork-lined holes in a block of wood. A simple bath for platinizing electrodes is also described.

J. H. MOORE.

Viscosimeter. Stormer. Drugs, Oils and Paints, 27 (1911); through Petroleum, 6, 2184.—For the detn. of the viscosity especially of paints and varnishes, this viscosimeter, manuf. by Bauch and Lomb, has been favored by several members of the committee D<sub>1</sub> of the Amer. Soc. of Testing Materials. A cylinder is made, by a special contrivance, to rotate, at a known temp., and under a constant wt., through the liquid under exam. The no. of revolutions are recorded by an instrument which has been calibrated with water.

O. E. Bransky.

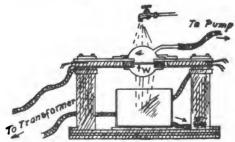
Stirring Apparatus for Heavy and Difficultly Miscible Liquids. H. Leiser. Chem. Ztg., 35, 756.—The 4 arms of the stirrer are of glass tubing, the lower ends flared and extending to different depths in the vessel. At a different height on each arm is an opening through which the liquid is discharged when forced up the tube by rotating the stirrer.

J. H. Moore.

A Tungsten Resistance Furnace for Chemical Uses. F. FISCHER AND E. TIEDE. Ber., 44, 1717.—As shown in the sketch, the app. consists of a small tube of W held

between the ends of 2 electrodes connected with a transformer for heavy current. The material to be heated is placed in a MgO boat in this tube and any temp., up to almost 3000° may be obtained. Pt, Fe, Ir, etc., may be substituted for the W. The heating element is surrounded by an evacuated, water-cooled glass bulb as shown.

W. E. RUDER.



Separating Apparatus, According to the Late Prof. Luedecke. O. DREIBRODT. Inst. Min. Petr., Univ. Leipzig. Contr. Min., 1911, 425-6.—Description, with figs., of a convenient app. for sepg. rock constituents with heavy solns. E. T. Wherey.

A New Electrical Testing Machine. G. KAPP. Elektrotech. Z., 34, 858.
W. E. RUDER.

Fusing Wires in Hydrogen (SOMERVILLE). 4. Electric Tube Furnaces (TUCKER). 4. Mercury Air Pump (BEUTELL). 8.

U. S., 1,000,403, Aug. 15. L. M. HANSEN, Upland, Nebr. Acetylene generator.

## 2. GENERAL AND PHYSICAL CHEMISTRY.

JOHN JOHNSTON.

J. H. van't Hoff. WILHELM OSTWALD. Ber., 44, 2219-52; see C. A., 5, 2206.—
Obituary.

J. J. M.

Alexander W. Williamson. G. C. Foster. Ber., 44, 2253-69.—Obituary.

J. J. M.

Stoichiometry. R. KREMANN. Fortschritte Chem., 4, 161-75.—Review for the period Dec., 1910 to July, 1911, under the main headings: general; vapor tension, etc.; Van der Waals' equation; mol. vol.; surface energy; viscosity; osmotic pressure.

J. **J**.

Theory of the Equation of State. C. DIETERICI. Kiel. Ann. Physik, 35, 220-42. J. J.

Internal Pressure in Gases: Equations of State and the Law of Molecular Attraction. A. Leduc. Compt. rend., 153, 179-82.—By calculations based on the author's equation of state (C. A., 3, 1234), the following law is derived: For gases of the same mol. constitution and at corresponding temps., the attraction between the mols. of the same gas is proportional to the square of their mass and inversely proportional to the fourth power of their distance.

Grinnell Jones.

Blue Color of the Sky and the Constant of Avogadro. E. BAUER AND M. MOULIN. Radium, 7, 372-3.—According to Rayleigh, the blue color of the sky is due to the dispersion of the light from the sun by the mols. of the air. By a comparison of the intensity of the light from the sky and from the sun the constant N may be calc. Earlier measurements were unsatisfactory since the 2 sources compared are of different color. Comparisons of the relative values of the intensity of the red H, yellow Na, and green Mg lines in the light from the sun and the sky were made by means of a spectrophotometer. The values of N varied from 10-50  $\times$  10<sup>-22</sup>.

MERLE RANDALL.

Comparison of the Platinum Thermometer with the Nitrogen, Hydrogen and Helium Thermometer, and the Determination of Certain Fixed Points between 200° and 450°. L. HOLBORN AND F. HENNING. Reichsanstalt, Charlottenburg. Ann. Physik, 35, 761-4.—In order to establish more definitly the temp. scale below 500°, measurements were made with various gas thermometers immersed in stirred liquid baths. At 200° an oil bath was used; at 330° and 450° a bath of fused KNO<sub>2</sub>. The thermometer bulbs were roughly of 300 cc. capacity, and were at first made of Jena glass 59<sup>III</sup> and filled with N, H, or He. Later, bulbs of quartz glass filled with N were

used. A comparison of several Pt thermometers gave values of δ in the Callendar formula ranging from 1.486 to 1.510. The value obtained for the b. p. of S on the thermodynamic temp. scale is 444.51° instead of 445.0° (Callendar, Chappuis and Harker). A recalc. of the previously obtained Pt temps. for the b. p.'s of naphthalene and benzophenone, using the new values of δ, gives 217.96° and 305.89° resp. The f. p.'s of Kahlbaum's Zn, Cd and Sn were newly detd. with the standardized Pt thermometers, with the following results: Sn, 231.83°; Cd, 320.92°; Zn, 418.40° (on the thermodynamic scale). These values agree closely with the Pt thermometer measurements of Callendar and Griffiths and of Waidner and Burgess when calc. on the basis of S b. p. 444.5°.

The Mechanical Viscosity of Fluids. T. E. STANTON. Nat. Phys. Lab. Proc. Roy. Soc. London (A), 85, 366-76.—The term "mechanical viscosity" has been used by Osborne Reynolds to designate the viscosity which arizes from the molar motion of a fluid and which is not a property of the fluid independent of its motion, as is its physical viscosity. The expts. by S. deal with the relation between shearing stress and rate of distortion in fluids which are in eddying motion, i. e., motion in which frictional resistance, at the boundaries of the solid over which they move, varies approx. as the square rather than the first power of the relative velocity. S. concludes that in general there is a central region in which the ratio of the shearing stress to the rate of change of distortion is const. for any particular value of the flow, and that in the neighborhood of the walls this alters rapidly until at the boundary it becomes equal to the physical viscosity.

L. H. A.

Specific Heat of Water. W. R. BOUSFIELD AND W. E. BOUSFIELD. Phil. Trans. Roy. Soc. London (A), 211, 200-53; Proc. (A), 85, 302-4 (abstract).—The object of this investigation was to obtain a basis curve for the sp. heat of water, for comparison with sp. heat curves of aq. solns. Former observers have obtained widely varying curves, and different values for the 15° cal. in terms of the joule. The authors have detd. the latter in terms of the mean cal. from 13-55° by a method of continuous flow calorimetry. Through a Dewar vessel containing 3 l. water, in which was an electric heater, there was passed a current of water entering at about 13° and passing out at about 55°; the temps. were read to 0.005° on Hg thermometers. The elec. heater, and resistances were of novel type. Each consists of a spiral glass tube of small bore into the ends of which are sealed Pt electrodes. The tube is connected with a thermometer tube so that the spiral forms a thermometer bulb. By calibrating the resistance against the reading of this thermometer tube, the resistance is accurately known, even when a current is passing. By this means the authors were enabled to surmount the difficulty that, when a heavy current passes through a standard resistance, the resistance depends not only on temp. but also on current strength. These expts. gave the value  $J_{13}^{86} = 4.182$ . To get the curve for J from  $0^{\circ}$  to  $80^{\circ}$ a weighed quantity of water was heated from o° to 80° by stages which gave  $J_0^{13}$ ,  $\int_{12}^{27}$ ,  $\int_{20}^{26}$ ,  $\int_{80}^{80}$ , the mean sp. heat over the intervals. From this series of expts. they derived the expression  $J = 4.2085 - 0.003022t + 0.00007833t^2 - 0.0000000490t^3$ which gives for the value of the 15° cal. 4.179 (Joule, 4174; Griffiths, 4.198; Barnes, 4.184). This curve corresponds closely with that obtained by Lüdin by the method of mixtures, but differs from that obtained by Barnes by continuous flow with Pt thermometry; it gives a minimum value of I about 25°. The values at 5° intervals follow: 0°, 4.209; 5°, 4.195; 10°, 4.186; 15°, 4.179; 20°, 4.176; 25°, 4.174; 30°, 4.175; 35°, 4.178; 40°, 4.182; 45°, 4.187; 50°, 4.192; 55°, 4.198; 60°, 4.203; 65°, 4.208; 70°, 4.213; 75°, 4.216; 80°, 4.217. J. JOHNSTON.

Experiments on the Compression of Liquids at High Pressures. C. A. PARSONS AND S. S. COOK. Proc. Roy. Soc. London (A), 85, 332-48.—The compressibility

of H<sub>2</sub>O, paraffin oil, Et<sub>2</sub>O and graphite was measured at pressures up to 6000 atm. The app. consisted of a steel mold of 4 in. bore and 12 in. external diam. Pressure was applied hydraulically on a tight-fitting plunger. The depression was measured by a pair of multiplying calipers properly attached to the plunger. Correction was made for errors due to friction and to compression of the packing. Expressing compressibility as the ratio of the decrease of vol. per atm. of pressure to the vol. of the liquid, the following values were deduced for the isothermal coeffs. of compressibilities (× 10°). H<sub>2</sub>O at 4°: 1 atm., 50; 2000 atm., 25; 4500 atm., 22.5. Et<sub>2</sub>O at 35°: 1 atm., 165; 1000 atm., 64; 2000 atm., 42.5; 4500 atm., 18. Paraffin oil at 34°: 1 atm., 87; 2000 atm., 34; 4500 atm., 17. The compressibility of graphite was about 6.5 × 10<sup>-6</sup>. The adiabatic expansion of liquids was also investigated. From the heat lost during such expansion, the following av. values of internal molar forces in atm., for pressures between 0 and 400 atms., were deduced: H<sub>2</sub>O, 2150; Et<sub>2</sub>O, 2440; paraffin oil, 2920.

Crystallization by Decompression. G. CRSARO. Bull. sci. acad. roy. belg., 1911, 436-7.—The surfaces of cylinders of Bi<sub>2</sub>S<sub>2</sub> and S which had been prepared some 10 yrs. earlier by compression were found to be covered with perfectly formed crystals. In the case of the Bi<sub>2</sub>S<sub>2</sub> the crystals, some of which were 0.1 mm. long, did not have the elongated forms of the natural crystals; they were about equally developed in all directions and had numerous finely glittering facets. The luster was adamantine rather than metallic. The crystals on the S were equally well defined but smaller (0.02-0.03 mm.). The author believes their formation was due to the slow expansion of the cylinders.

The Modulus of Elasticity and Thermal Expansion of Metals. H. SIEGLERSCHMIDT. Ann. Physik, 35, 775-82.—The empirical formula,  $(E/S)/34000 (1/A\beta)^{1.2} = 1$ , is shown to connect the elastic and thermal properties of metals better than the formula he has previously proposed. In this formula  $\beta$  is the coeff. of expansion, E the modulus of elasticity in g./cm.<sup>2</sup>, E the density, and E the at. wt.

The Latent Heat of Fusion and the Specific Heat of the Fatty Acids. G. Massor And A. Faucon. Compt. rend., 153, 268-70.—For method and previous work see C. A., 3, 2650. The mean sp. ht. of solid lauric acid (m. p. 43.62°), is 0.388 at —22.4°, 0.430 at 0°, 0.540 at 34.8°. The sp. ht. of the liquid at 57.25° is 0.515. The latent ht. of fusion is 46.68 cal., the latent ht. of solidification, 37.42 cal. The acid used was pure, congealed rapidly to a cryst. mass; the temp. equil. was rapidly attained and remained constant. Impurities change the appearance of the crystals and increase the time required for equil. The authors explain the anomaly by assuming the solid to be a mixt. of the cryst. and the amorphous substance.

George W. Morrey.

Properties of Binary Mixtures of Some Liquefied Gases. LANCELOT S. BAGSTER. Univ. Melbourne. J. Chem. Soc., 99, 1218-23; Proc., 27, 141; see C. A., 5, 1015.—The systems, H<sub>2</sub>S-HI and H<sub>2</sub>S-HBr, have now been studied further by detg. their f. p. curves. These latter are of the same general shape; each shows a minimum, but neither is of the characteristic shape with 2 eutectic points and a max., exhibited by a mixt. in which compd. formation takes place. Their form is that which is produced by a system in which the solid phase consists of mixed crystals. The HBr-H<sub>2</sub>S system may then be regarded as adducing further evidence in favor of the conclusion of Tsakalotos (C. A., 4, 1402) that a minimum in the vapor pressure curve of a binary mixt. does not necessarily indicate the formation of a compd.

L. H. Adams.

The Effect of Temperature and Pressure on the Equilibrium: 2 CO = CO<sub>2</sub> + C. THOMAS F. E. RHEAD AND RICHARD V. WHEELER. Altofts. J. Chem. Soc., 99, 1140-53; Proc., 27, 128.—The authors have previously investigated this equil. at atmospheric

pressure  $(C.\ A., 5, 641)$ . Further measurements have now been made at 0.5-3 atm., in order to prove whether the expression (N'-N)  $ln\ P$  in Le Chatelier's general equation for mobil equil. satisfies expt. in this particular case. The reaction vessel of "quartz" glass was heated in an elec. resistance furnace wound with Pt wire. Temps. ranged from  $800^{\circ}$  to  $1100^{\circ}$  and were measured by means of a Pt-PtRh thermocouple placed outside the quartz vessel. The equation  $(38,055+2.02\ T-00031\ T^2)2\ T+lm\ P+lm\ (C_1^2/C_2)=k$  was found to satisfy the data obtained. In this equation T is abs. temp., P the pressure in atm.,  $C_1$  and  $C_2$  the fractional cones. of CO and CO<sub>2</sub> resp., and k a const. Equil. was reached rapidly at the higher temps., but at  $800^{\circ}$  it was attained only after heating for more than 70 hrs.

Remarks on Some Experiments of M. Palladino. G. VAN DER MENSBRUGGHE. Bull. sci. acad. roy. belg., 1911, 561-2.—The observation of Palladino (Riv. fis., math. i sci. nat., Pavia, 1910, Sept.) that if the 2 arms of a glass capillary U-tube are of unequal length or if one is drawn out to a fine point slightly volatil liquids will not rize to the same height in both arms is in harmony with the numerous facts brought out by the author to show that there is a force of extension at the surface of sepn. between glass and H<sub>4</sub>O.

Chas. A. Rouiller.

The Direct Measurement of Vapor Pressure Changes and the Detection of Compounds by Vapor Pressure Changes as a Lecture Experiment. RUDOLF KRULLA. Chem. Ztg., 35, 471-2.—Through the stopper of a flask immersed in a thermostat passes an airtight stirrer, the tip of a buret, and a tube connecting with the top of the buret and with a manometer. The flask contains one liquid component; the other is run in in measured amt. from the buret, and after equil. is reached the pressure read on the manometer. Curves so obtained showing the relation between v. p. and comp. are given for the pairs CCl<sub>4</sub> and PhNH<sub>2</sub> and PhNH<sub>2</sub>-PhOH, the compd. PhOH.PhNH<sub>2</sub> being indicated in the latter case.

George W. Morey.

Some Objections Made Recently by A. Colson to the Ionic Theory. G. URBAIN. • Bull. soc. chim., [4] 9, 753-8.—A criticism of Colson's paper (C. A., 5, 3187), pointing out that Colson's results do not conflict with the ionic theory.

J. JOHNSTON.

Theories of Solutions. James Walker. Chem. News, 104, 104-8; Chem. Trade J., 49, 241-3.—W. considers especially the case of strong electrolytes and brings forward cogent evidence in favor of the view that their abnormal behavior is to be attributed to the non-ionized portion rather than to the ions. "To put the whole matter briefly, in the equil. between electrolytes agreement will be obtained between theory and expt. whether we use the mass-action law, or an empirical law such as van't Hoff's dilution formula, provided only that we attribute the abnormality to the non-ionized portion of the electrolyte. Thus we can deduce the ordinary formulas for hydrolysis or for isohydric solns. as readily for abnormal as for normal electrolytes, and find the most satisfactory agreement with expt. in both cases."

Studies on Chemical Affinity. V. Formation of Potassium Lead Sulfate. J. M. BRÖNSTED. Tech. High School, Copenhagen. Z. physik. Chem., 77, 315-30; cf. C. A., 3, 604; 4, 1415, 2399; 5, 3364.—The conc. of K<sub>2</sub>SO<sub>4</sub> at which the double salt is in equil. with both soln. and PbSO<sub>4</sub> was found = 0.0227 mol. at 22°; 0.0112 mol. at 0°. These values are much lower than those of Barre (C. A., 3, 2781). The formula of the double salt is K<sub>2</sub>SO<sub>4</sub>.PbSO<sub>4</sub>. This salt is always pptd. instead of PbSO<sub>4</sub> if more than 0.4% K<sub>2</sub>SO<sub>4</sub> is present, and, in presence of other sulfates or H<sub>2</sub>SO<sub>4</sub>, if the conc. of K salt is still smaller. The free energy of formation was detd. from e. m. f. measurements, using the chain, Pb PbSO<sub>4</sub> |Hg<sub>2</sub>SO<sub>4</sub>|Hg|Hg|Hg<sub>2</sub>SO<sub>4</sub>/K<sub>2</sub>SO<sub>4</sub>.PbSO<sub>4</sub>,K<sub>2</sub>SO<sub>4</sub>|Pb. In the first element PbSO<sub>4</sub> was formed from Pb-amalgam and Na<sub>2</sub>SO<sub>4</sub> of different concs. Its e. m. f., which became constant after a few hours, is independent theoretically,

of the conc. A series of researches established this independence at lower temps., at higher temps, the dil. solns. showed decreased temp. change of e. m. f. Found  $\pi = 0.0561 - 0.000037(t - 22)$ . The K<sub>s</sub>SO<sub>4</sub> element was prepared with PbSO<sub>4</sub> as well as with the double salt. In both cases the e. m. f. was established very slowly and increased for several days. Elements in which the prepared double salt was used reached constant e. m. f. after 3 or 4 days. By dividing into 2 half elements, reaction between  $K_2SO_4$  and  $Hg_2SO_4$  was found absent. Found,  $\pi = 1.0481 - 0.000100$ (t-22). The difference between the reaction in the K<sub>2</sub>SO<sub>4</sub> element, Pb + Hg<sub>2</sub>SO<sub>4</sub> + K,SO, ---> K,SO, PbSO, + 2 Hg, and that in the Na,SO, element, Pb + Hg,SO, ---> PbSO<sub>4</sub> + 2 Hg, or  $K_1SO_4$  + PbSO<sub>4</sub>  $\longrightarrow$   $K_2SO_4$ . PbSO<sub>4</sub> is the affinity coeff.,  $\pi = 0.0920$ -0.000063(t-22) or in cal. per mol., A = 4240-2.9(t-22). For the soln. affinity (cf. C. A., 4, 2399), the author deduces the equation,  $A_1 = -291.0[6.841]$  $\log c - 1.278 (\log c)^2 - 0.0888 (\log c)^2 + k$ . Substituting in this, first the satn. conc., c = 0.423, and then the equil. conc., c = 0.0112, 2 values of  $A_1$  are obtained, 782 + k and 5109 + k, the difference of which, 4327 cal., is the formation affinity of 1 mol. at 0°. For the heat of formation of the double salt  $U = 5310 \pm 200$ cal. was obtained as the mean of 2 calorimetric series; calc. from the value for A found by e. m. f. measurements, U = 5000 cal. The latter is probably more accurate. The affinity of the double salt formation decreases with rizing temp. similarly to that of PbCl<sub>2</sub>.2 NH<sub>4</sub>Cl (C. A., 5, 3364). Refuting criticisms of Thomsen's thermochem. data for Pb, the author points out that Klein's e. m. f. detns. of the heat of formation of PbSO, and Pb halides (Z. physik. Chem., 36, 361) are in error through the presence in his elements of PbSO, K,SO,. Recalculating Klein's data upon this basis the agreement with the thermochem. data is within the error of expt.

A. R. MIDDLETON.

The Hydrolysis of Iodine and of Bromine. A Correction. W. C. BRAY AND E. L. CONNOLLY. Mass. Inst. Tech. J. Am. Chem. Soc., 33, 1485-7; cf. C. A., 4, 2758. —On account of the presence of  $I_2$  and  $Br_2$  the values of the hydrolysis constants should be  $0.3 \times 10^{-12}$  and  $5.2 \times 10^{-9}$  resp.

A. R. M.

Transference Numbers and Molecular Complexity. ARRIGO MAZZUCCHELLI. chim, r. univ, Roma. Atti accad. Lincei, 20, II, 124-9.—Suppose a weak, polymerized acid in conc. soln. on dissociation gives a single H+ ion, then if its degree of polymerization is represented by a and its minimum equiv. wt. (detd. by chem. means) by e, by the Stokes-Einstein formula the mobility of the anion is theoretically  $l_a = vF/$  $6\pi\eta N \sqrt{3as/4\pi SN}$ , where F is the force to which I g. of a monovalent ion is subjected in an elec. field of intensity 1 v. per cm., v the valence, N the no. of mols. in 1 g. mol., 7 the coeff. of viscosity of the liquid, and S the d. of the mol. Experimentally,  $l_a =$  $l_{cg}/(Qae - g)$ , where  $l_{c}$  is the mobility of the cation, g the wt. of anion which has migrated and Q the equivs. of Ag deposited in the voltameter. Equating the 2 expressions for  $l_a$  in terms of a, an expression of the form  $GDa - Dg = La^{1}$  is obtained which can be solved for a. In the other limiting case, vis., where the electrochem. equiv. of the anion is known but its valence unknown (e. g., an alk. salt of a polymerized acid in which the cations all dissociate simultaneously), a must be substituted for v in the first expression for  $l_a$  given above, and the second becomes  $l_a = l_c n/(1-n)$ , where n is the transference no. of the ion. Equating the 2 equations, an expression of the form  $A = Ba^{2/2}$  is obtained. CHAS. A. ROUILLER.

The Elimination of Diffusion Potentials in the Measurement of Electrode Potentials. NIELS BJERRUM. Univ. Copengahen. Z. Elektrochem., 17, 389-93.—B. measured the potential of 9 cells composed of HgCl electrodes with solns. of HCl, KCl, or NaCl, the liquid contact being made in layers of sand under conditions to which the Henderson formula for the diffusion potential applies instead of the Planck formula (see C.

A., 1, 2766; 2, 2893; 5, 2768). It is shown that the insertion of a satd. (3.5 N) KCl soln. greatly reduces the diffusion potential and that the remaining diffusion potential is made up of two parts due to (1) difference between the mobility of K<sup>+</sup> and Cl<sup>-</sup>, (2) the effect of the ions in the dilute soln. By inserting half satd. KCl instead of satd. KCl, (1) remains the same but (2) is twice as great and, therefore, the error due to (2) can be eliminated by a simple extrapolation. The error due to (1) is small, if the two solns. around the electrodes are of the same conc., but becomes 0.0007 v. between 0.1 N KCl and 0.001 N NaCl. Except in unfavorable cases the extrapolated result is reliable to 0.0005 v. NH<sub>4</sub>NO<sub>2</sub> may be used instead of KCl, but is not as good owing to the greater difference in the mobility of the ions. (The results show that it is better to make the liquid contact in sand without adding any other soln. and eliminate the diffusion potential by calc. by Henderson's formula. ABSTR.)

GRINNELL JONES.

Rectromotive Forces in Alcohol. I. Concentration Cells with Electrodes Reversible to Chlorine Ions. A. Lapworth and J. R. Partington. Univ. Manchester. J. Chem. Soc., 99, 1417-27; Proc., 27, 194.—The work described in this paper was begun in connection with the study of equil. between electrolytes in alc., and in particular of the equil. between mineral acids, EtOH and H<sub>2</sub>O. The e. m. f. of the conc. cells Hg | Hg<sub>2</sub>Cl<sub>2</sub> | (HCl-EtOH)<sub>1</sub> | (HCl-EtOH)<sub>2</sub> | Hg<sub>3</sub>Cl<sub>2</sub> | Hg and Ag | AgCl | (HCl-EtOH)<sub>1</sub> | (HCl-EtOH)<sub>1</sub> | (HCl-EtOH)<sub>2</sub> | AgCl | Ag, which appear to fulfill the conditions for reversibility, and as a rule give nearly constant values at 25°, have been studied. From these e. m. f.'s the transport number of Cl<sup>-</sup> in EtOH is calc. to be about 0.37, contrasted with 0.16 in H<sub>2</sub>O. Conds. of solns. of HCl in EtOH at 25° varying in conc. from 0.634 N to 0.001025 N were measured and by extrapolation I<sub>0</sub> was calc. to be 66.5.

Investigation of Silicates. ARTHUR L. DAY. Z. Elektrochem., 17, 609-17.—A brief review of the investigations of silicates carried out in the Geophysical Lab. of the Carnegie Institution of Washington.

J. J.

Cryoscopic Study of Some Mineral Acids and Phenols. EUGENE CORNEC. Compt. rend., 153, 341-3; cf. C. A., 4, 701, 725.—In this paper the author confirms the formulas HClO<sub>3</sub>, HClO<sub>4</sub> and HIO<sub>5</sub>.  $C_6H_5OH$  acts like a monobasic acid.  $H_7SeO_2$  shows a minimum with the neutral salt; the acid salts correspond to the formula MHSeO<sub>3</sub>. The formula  $H_7S_3O_6$  is confirmed, the minimum being at the neutral salt. Neutral salts of  $H_7CO_2$  exist in soln. Resorcinol shows two replaceable H atoms. The formula  $H_4P_3O_7$  is confirmed, the minimum in this case corresponding to the neutral salt. With  $H_7S_2O_3$  only the first  $H_7^+$  is indicated. The formula  $H_2BO_3$  is confirmed, though its behavior is that of a monobasic acid. The insufficient lowering observed indicates the formation of acid salts, though the curve shows no break. Mixts. of sucrose and NaOH solns. in all proportion show a similar curve, probably due to the formation of Na sucrate.  $H_2ASO_3$  resembles  $H_3BO_3$ , acting like a monobasic acid. Curves of a third type were obtained with  $H_2CrO_4$  and  $HIO_4$ , indicating some phenomenon other than neutralization.

Thermal Analyses of Binary Mixtures of Chlorides of Bivalent Metals. C. Sandonnini and G. Scarpa. Lab. chim. gen. r. univ. Padova. Atti accad. Lincei, 20, II, 61–8.—The following f. p.'s were used: SnCl<sub>2</sub>, 250°; CdCl<sub>2</sub>, 568°; PbCl<sub>2</sub>, 495°; MnCl<sub>3</sub>, 650°. The systems SnCl<sub>2</sub>-PbCl<sub>2</sub> and CdCl<sub>2</sub>-MnCl<sub>2</sub> form mixed crystals in all proportions; CdCl<sub>2</sub>-SnCl<sub>3</sub>, MnCl<sub>3</sub>-SnCl<sub>4</sub> and MnCl<sub>3</sub>-PbCl<sub>2</sub> give simple eutectics for 90 mol. % of SnCl<sub>3</sub> at 233°, 95% SnCl<sub>4</sub> at 233° and 70% PbCl<sub>4</sub> at 408°, resp. Chas. A. ROUILLER.

Aluminium Bromide as a Solvent. B. A. ISBEKOV AND V. A. PLOTNIKOV. Polytech. Inst., Kiev. Z. anorg. Chem., 71, 328-46.—The authors state that fused AlBr<sub>2</sub>,

on account of its low in. p., the fact that it dissolves a large number of salts and when pure is a nonconductor, serves as an admirable solvent for the study of the interaction of solvent and solute and the ionization of fused salt mixts. KBr, NaBr, NH,Br, AgBr, CaBr, dissolve in AlBr, at about 100° with the formation of 2 layers, the upper containing only a small amt. of the dissolved salt. On further addition of salt the upper layer gradually disappears until at a conc. of about 11% all of the above salts form homogeneous solns. The bromides of Cu, Zn, Cd, Hg, Bi, Mn, Fe and Co, give completely homogeneous solns.; those of Zn, Hg and Bi, up to a conc. of about 20 or 30%, the others to a less extent. The solubility increases markedly with a rise in temp. SnBr, SbBr, and AsBr, are miscible with AlBr, in all proportions. BaBr, NiBr, CuBr, and Al<sub>2</sub>O<sub>3</sub> are practically insol. HgBr at 220-230° dissolves to the extent of 3 or 4%. CBr4 and CBr4 dissolve well in AlBr2, but do not conduct the current, while a soln. of dimethylpyron C<sub>2</sub>H<sub>2</sub>O is a very good conductor. The sp. conds. of solns. of KBr, HgBr, and SbBr, of various concs. are tabulated and plotted. The measurements were made about 100°, the cond. cell being entirely surrounded by steam. The measurements on KBr and HgBr cover only a small range of conc. In the case of SbBr<sub>2</sub> the concs. extend from 100% AlBr<sub>2</sub> to 100% SbBr<sub>2</sub>. Neither of these substances when pure will conduct the current, but mixts. of the 2 are good conductors. The cond. shows a decided max. at 81% SbBr, which corresponds well with a comp. AlBr, 3 SbBr<sub>2</sub>. The influence of temp. on the cond. of solns. of 7.81%, 14.38% and 21.08% HgBr<sub>2</sub>; 15.27% and 50.35% SbBr<sub>2</sub>, and 13.07% KBr was measured. The cond. was measured at intervals of 10° between 99.5° and 190.1°. The cond. of the 7.81% and 14.38% HgBr, and the 15.27% SbBr, solns. increases to a max. and then falls off rapidly; that of the other 3 solns. increases continuously with rise in temp. By electrolysis with Pt electrodes Br was liberated at the anode in all cases and at the cathode Sb from the SbBr<sub>2</sub> soln., Hg from the HgBr<sub>2</sub> soln., and Al from the KBr soln. The amt. of Sb deposited by a given current was found to be only 74.5% of the theoretical amt. The d<sup>99,5</sup>° of AlBr<sub>2</sub> is 2.754, of SbBr<sub>2</sub>, 3.845; those of mixtures of the two and mixtures of HgBr, and KBr with AlBr, are also given. The interaction of solvent and solute and not "the dissociating power" of the solvent conditions the cond. of these mixts., since solns. of AsBr<sub>2</sub> and SnBr<sub>4</sub> do not conduct the current at all, while solns. of most of the other salts are good conductors. J. L. CRENSHAW.

Definit Compounds with Varying Composition of the Solid Phase. I. Conductivity and Hardness of the System: Magnesium-Silver. V. I. SMIRNOV AND N. S. KURNAKOV. Polytechn. Inst., Univ. St. Petersburg. Z. anorg. Chem., 72, 31-54.—The expts. described in this paper were undertaken with the hope that they would throw light on the question of the definition of a chem. individual and on the whole subject of solid solns. The general types of diagrams for the cond. and hardness for definit chem. compds. with variable comp. of the solid phase are shown. A study of the cond. in the case of the system Mg-Ag indicates that the substances which are formed in this system exhibit a variable comp. of the solid phase. In the case of the solid solns. of MgAg there was observed, in accordance with theory, a max. which lies at the intersection of the 2 descending branches of the diagrams of elec. cond. and of the temp. coeff. This point corresponds to an at. ratio Mg: Ag = 1: 1. Investigation of hardness by the method of Brinel showed, for the solid solns. of MgAg, a minimum at 50 at. % Ag. The temp. coeff. of elec. resistance is a very important and characteristic quantity. The numerical values for the argentides MgAg and MgAg are similar to those of the pure metals. This observation is in complete agreement with the results of N. Stepanov for other metals. L. H. ADAMS.

The Recognition of Racemic Substances. H. R. KRUYT. van't Hoff Lab., Utrecht. Ber., 44, 995-9.—Polemical (cf. C. A., 5, 1907). The author asserts that

the results of Ladenburg not only do not contradict Roozeboom's assertion that the phase rule applies to stereomers, but also supports that assertion. Further, Ladenburg's results disprove the existence of liquid racemic compds. George W. Morey.

The Application of the Phase Rule to Stereomeric Compounds and the Recognition of Racemic Substances. T. VAN DER LINDEN. Univ. Amsterdam. Ber., 44, 963-5.—Polemical (cf. C. A., 5, 1907 and preceding abstr.). The author concludes that the question of the existence and identification of liquid racemic compds. is not settled. He shows that the method of Ladenburg for the identification of dl-compds. is not applicable, while the phase rule adequately explains all facts observed with stereomeric mixtures.

G. W. M.

Application of the Phase Rule to Stereochemistry and the Recognition of Racemic Compounds. A. LADENBURG. Ber., 44, 1677; cf. preceding abstrs.—The author maintains that his rule for the detection of dl-compds. is applicable to liquids.

J. BISHOP TINGLE.

The Influence of Foreign Substances on the Activity of Catalyzers. C. PAAL AND A. KARL. Pharm.-Chem. Inst., Univ. Erlangen. Ber., 44, 1013-8.—Mg, Al, Fe, Ni, Co, Cu, Zn, Ag, Sn and Pb powders coated with Pd were prepd. by digesting the cleaned metal in PdCl<sub>2</sub> soln., washing with EtOH and Et<sub>2</sub>O and drying in vacuo. The value of the Pd coated metal powders as carriers of H was studied, using a liquid, unsatd. ester, and the app. of Paal and Gerum (C. A., 2, 1792). Mg, Ni and Co have no effect on the activity of Pd. The other metals all act as anticatalysts. The effect of solvents on the action of the Pd was studied with the active Pd-coated Ni powder, the unsatd. ester being dissolved in various solvents. C<sub>2</sub>H<sub>2</sub> and Me<sub>2</sub>CO are both anticatalysts, Et<sub>2</sub>O and EtOH are without effect. The poisoning action of C<sub>2</sub>H<sub>4</sub> was such that the activity of the Pd-coated Ni could not be restored by treatment with Et<sub>2</sub>O or by gentle ignition in H.

Extension of the Laws of Capillarity to the Case where the Elements of the Capillary Systems are Mobil with Respect to each other; Giving a New Conception of the Phenomenon of the Inflation of Dried Wood, of the Dissolving of Gums, 'Albumins, etc., and of True Solutions. J. H. Russenberger. Compt. rend., 153, 57-60.—The author suggests that the phenomenon of the swelling or soln. of porous substances in presence of liquid can be attributed as well to the known laws of capillarity as to osmotic effect.

George W. Morey.

Colloidal Iron Hydroxide. A. DUMANSKII. Kiew. Z. Chem. Ind. Kolloide, 8, 232-3.—Colloidal Fe hydroxide was prepared by dialysis of a soln. of FeCl<sub>e</sub> satd. with  $(NH_4)_2CO_3$ , and then allowed to stand 1 year. The soln. so prepared was filtered through collodion and the mol. wt. of the colloidal particles calc. from the differences in f. p. of the filtrate and the residual soln., and found to be 3122(n+1) where n represents the number of ions in equil. with the colloidal particle. The d. of the colloidal particle (x) was calc. from the formula  $A = B + C - C/x\delta$  (J. Russ. Phys. Chem. Soc., [2] 35, 585 (1903); [2] 38, 129 (1906)) where <math>A = wt. of soln., B = wt. of solvent, C = wt. of colloid and  $\delta =$  d. of filtrate; it was found to be 4.704 at 0°. From this the author concludes that the colloid is a mixture of oxide and chloride. H. ISHAM.

The Dissymetry of Positive and Negative Ions Relative to the Condensation of Water Vapor. E. Besson. Compt. rend., 153, 250-3.—The author repeats the expts. of C. T. R. Wilson (Phil. Trans., 193, 1899) using a photographic method. His expts. show plainly the greater condensation with preponderance of negative ions, confirming the results of Wilson.

George W. Morey.

Absence of the Influence of Light on the Conductivity of Fluorescent Solutions. S. PIENKOWSKI. Inst. phys. inst. Liège. Bull. sci. acad. roy. belg., 1911, 594-602.

-Expts. on eosin and fluorescein in H<sub>1</sub>O, alc. or gelatin showed that there is no change in cond. when the soln. is illuminated by white or monochromatic light. Various forms of app. were devized to prevent complications due to heating, polarization, etc.

Chas. A. ROUILLER.

Influence of Light on the Conductivity of Thin Sheets of Gold and Silver. S. Pienkowski. Inst. phys. univ. Liège. Bull. acad. roy. belg., 1911, 603-4.—Light has no influence on the cond. of transparent deposits of Au and Ag on glass. Negative results were likewize obtained with the identical Au layer used by Bronislavski (C. A., 3, 1958) who states that white light doubles and blue light increases 75% the cond. of the deposit.

C. A. R.

The Intensity of the Ultraviolet Light Emitted by an Electrical Discharge at Low Pressures. E. W. B. Gill. Phil. Mag., 22, 412-9.—A discharge was produced in air contained in a quartz tube between Al electrodes 7.5 cm. apart, and the intensity of light proceeding from various parts of the discharge, estimated by the rate at which a Zn plate lost its charge. The max. light intensity is near the anode for pressures exceeding that corresponding to minimum spark potential; for pressures less than this it lies near the cathode.

ROBT. F. EARHART.

The Coefficient of Magnetization of Gold. M. Hanriot and F. Raoult. Compterend., 153, 182-5.—One of the authors has previously noted that when a Ag-Au alloy is parted with HNO3, a brown Au remains, which he believes to be a new form of Au, Au $\beta$ . A number of deths. of the coeff. of magnetization gave irregular results for both  $\alpha$ Au and the  $\beta$ Au obtained from it by calcination, though the difference between the 2 was always evident. More regular results were obtained after successive treatments with HNO3 to remove Fe, the coeff. increasing for both forms. The transition of  $\beta$ Au into  $\alpha$ Au does not take place below 250°, commences at 300°, and is complete above this temp.

George W. Morey.

The Hysteresis Loss of the Ferromagnetic Manganese-Aluminium Bronzes in Relation to the Frequency of the Alternating Field. Desider Steiner. Techn. Hochs. Darmstadt. Ann. Physik, 35, 727-54.—Comparative measurements of the hysteresis loss and eddy current loss of Heusler alloys and Fe showed that these alloys behave in this respect similarly to Fe. The hysteresis loss increases and the eddy current loss decreases, as does that of Fe, with increasing frequency of the magnetizing current. The frequencies employed ranged from 2000 to 5000 per sec.

L. H. Adams.

Osmosis of Electrolytic Solutions (GIRARD). 11.

### 3. RADIOACTIVITY.

### HERMAN SCHLUNDT.

The Corpuscular Ionization of Saline Vapors and the Recombination of the Ions of a Flame. G. Moreau. Compt. rend., 152, 1664-6.—The increase in cond. previously observed to take place between Pt electrodes suspended in a flame charged with an alk. salt, when the cathode was covered with a layer of CaO (C. A., 5, 3008), was likewize found to occur when the cathode is covered with BaO, SrO, or any alk. salt. In every case the cond. of the vaporized salt is analogous to that of an ordinary ionized gas. The current increases with the strength of field and tends towards the satn. current S which, however, is not experimentally attained, but may be calc. from observations made with fields of low intensity. With intense fields the number of corpuscles which returns to the cathode is insignificant, and consequently the number

of salt mols. p which each cathodic corpuscle is able to dissociate is given by the equation,  $p = Q/Q^1$ , where  $Q^1$  is the observed current. With the same cathode, p does not vary with the acid radical of the salt vaporized, and varies but little with the metal radical. The value of p, however, changes with the nature of the cathode, its temp., and the duration of heating. At a temp. of about  $1500^\circ$  it was found that p does not exceed 20 for CaO, or 10 for SrO, BaO, or Na<sub>2</sub>CO<sub>3</sub>. With different cathodes and various saline vapors the coeff. of recombination of ions was found to vary between 5,000 and 20,000 e. s. u. It follows from this that recombination occurs once in about 500 collisions which is less rapid than the rate of recombination that takes place in ordinary air.

The Influence of Various Physical Conditions on the Ultraviolet Radiation of Quartz Mercury Vapor Lamps. Victor Henri. Compt. rend., 153, 426-9.—By using the citrate of Ag method described in a previous article, a study was made of the variation in intensity of the ultraviolet rays from a Westinghouse Cooper-Hewitt lamp when subjected to varying conditions of cooling. The ultraviolet radiation of the lamp is more intense as the temp. of the luminous tube increases. With the tube cooled in water the ultraviolet rays evolved are only 1/14 as intense as when the lamp is in air, the watts consumed remaining the same in both cases. A comparison of 6 lamps of different makes and ages showed that their ultraviolet radiations differ but little from one another, and that the quartz Hg vapor lamp thus constitutes a const. source of rays for photochemical and other expts. involving the use of ultraviolet rays, the radiation being defined when the voltage, amperage and length of the tube are known.

W. H. Ross.

The Probable Influence of the Soil on Local Atmospheric Radioactivity. J. C. Sanderson. Sloane Physic. Lab., Yale Univ. Am. J. Sci., 32, 169-84.—A detn. was made of the amt. of Ra Em. in the underground air in the neighborhood of the Sloane physic. lab. It was found that 1 cc. of underground air contains Ra Em. equal to the amt. in equil. with  $2.4 \times 10^{-13}$  g. of Ra. This is equiv. to the production, per cc. of earth, of Ra Em. in equil. with  $8.9 \times 10^{-14}$  g. of Ra. A method has been devized by which the amt. of Th Em. present in underground air can be directly measured in an electroscope and compared with the Em. evolved by a known wt. of Th salt. In this manner it was found that the earth in the neighborhood of this lab. emits per cc. Th Em. equiv. to that produced by  $1.35 \times 10^{-6}$  g. of Th under the standard conditions. The important bearing of the emanating power of the soil on the radioactive properties of the atmospheric air in different localities was pointed out, and a method was suggested by which the emanating power of the soil and the radioactive character of the underground air could be detd. in any given locality. W. H. R.

An Extremely Absorbable Ionizing Radiation Emitted by Radium C. Louis Wertenstein. Compt. rend., 152, 1657-60.—The relatively intense ionizing radiation with penetrating power analogous to that of radioactive projections (cf. C. A., 4, 1843; 5, 26) which is given off from a deposit of Ra C appears to be produced by the projection of Ra D from Ra C, since the intensity of the radiation is always proportional to the amt. of Ra C on an active disk, and it is only slightly deviated by a magnetic field.

W. H. R.

The Action of Radium Emanation on Thorium Salts. M. HERSCHFINKEL. Compt. rond., 153, 255-7.—The author repeated the expts. of Ramsay and Usher (C. A., 4, 991) on the formation of CO<sub>2</sub> through the action of Ra Em. on (ThNO<sub>2</sub>)<sub>4</sub> by using a special app., to be described more in detail elsewhere, in which all contamination from grease on the stopcocks could be completely avoided. The Th(NO<sub>2</sub>)<sub>4</sub> used was carefully purified to remove all organic impurities, but when 125 g. of the salt were exposed in soln. for 12 days, the CO<sub>2</sub> which formed in the soln. amtd. to 1 cc. When the Th

soln. was replaced with pure water only a very small amt. (o. 1 cc.) of CO<sub>2</sub> was obtained, and when the Th soln. was allowed to stand for the same length of time without being exposed to the Em. only a trace of CO<sub>2</sub> was formed, but when a small amt. of KMnO<sub>4</sub> was added, a larger quantity of CO<sub>2</sub> was obtained than when the Th soln. was exposed to the Em. It is therefore concluded that the CO<sub>2</sub> obtained in a soln. of Th(NO<sub>2</sub>)<sub>4</sub> exposed to the Em. does not come from a transformation of the Th into C as stated by Ramsay and Usher, but results from the oxidizing action of the Em. on some organic impurities, probably oxalic acid, in the Th(NO<sub>2</sub>)<sub>4</sub> soln.

W. H. R.

Action of Niton (Radiation Emanation) on Thorium Salts. SIR WILLIAM RAMSAY. Compt. rend., 153, 373-4.—In reply to the criticism of Herschfinkel (see preceding abstract) the author states that the quantity of Th(NO<sub>2</sub>)<sub>4</sub> exposed to Ra Em. in the expts. of Ramsay and Usher was not as great as stated by Herschfinkel; that the small quantity really exposed to the Em. was first freed from organic impurities by igniting to redness after which the residue was dissolved in HNO<sub>2</sub>, and crystallized several times in a dust free atm.; that the Em. in equil. with 0.6 g. of RaBr, to which the soln. of the pure crystals obtained were exposed, had been carefully freed from CO<sub>2</sub>, and had not come in contact with either greases or rubber; that CO<sub>2</sub> was always obtained when Th(NO<sub>2</sub>)<sub>4</sub> was exposed to the Em. but not when the nitrates of Bi, Hg or Ag were used instead; and that the expts. of Herschfinkel only prove that the Th(NO<sub>2</sub>)<sub>4</sub> used by him contained something which yielded CO<sub>2</sub> when treated with KMnO<sub>4</sub> which ordinarily of itself evolves a considerable amt. of CO<sub>2</sub> when ignited.

W. H. R.

The Molecular Weight of Thorium Emanation. MAY SYBIL LESLIE. Compt. rend., 153, 328-30.—The app. devized by Debierne (C. A., 4, 3163) for detg. the mol. wt. of Ra Em. by diffusion through a small opening was used by the author with some modifications to det. the mol. wt. of the Th Em. As a result of a number of expts. in which the rate of diffusion of the Th Em. was compared with that of O, it was found that the mol. wt. of the former must be in the neighborhood of 200. W. H. R.

The Beta Rays of the Radium Group. J. Danysz. Compt. rend., 153, 339-41. —An investigation was made of the comp. of the  $\beta$ -rays from the Ra group by adopting under improved conditions the method first used by Becquerel which consisted in photographing the trajectories of the rays when acted upon by a magnetic field. The source of the rays consisted of Ra Em., from 0.12 g. of RaCl<sub>2</sub>, which was condensed in a small glass tube with walls only 0.04 mm. in thickness. In this way the interference of secondary rays was avoided. The magnetic spectrum obtained showed the existence of seven distinct homogeneous beams of rays. The velocity of the different rays were calculated to be approx., 0.98, 0.95, 0.86, 0.81, 0.75, 0.69 and 0.63, the velocity of light being taken as unity.

The Coloring of Inorganic Substances by Radium. NIEDERSTADT. Elektrockem. Z., 18, 131.—Beta rays give interesting, lasting colorations, especially with the alk. halides. A short review is given of the effect of the various rays on different minerals.

W. E. Ruder.

Age of the Earth. J. Joly. Phil. Mag., 22, 357-81.—The lapse of time since the oceans were formed and stratification of rock began may be reckoned in 3 ways. (1) By estg. the Na content of the ocean and the amt. carried annually by rivers to the ocean. The age of the earth is then 80-150 mil. yrs. (2) Assuming an av. accumulation of 3 in. of sediment per century, the estimate is 148 mil. yrs. (3) A third method is based on radioactive changes. Since 1 g. of U gives rise to 10.7 × 10<sup>-8 cc</sup> of He per yr., the He content of the older formations of stratified rock will give a value (715 mil. vrs.) for the age of the earth. A second radioactive method, based on the accumulation of Pb, gives 1640 mil. yrs. The validity of the assumption that there is a constant change of the parent radioactive substances is questioned. ROBT. F. HARHART.

The Disintegration Products of Uranium G. N. ANTONOFF. Phil. Mag., 22, 419-33.—When U-X is sepd. by a Ba method (1) and by a ferric method (2), the rate of decay of the unscreened product is not the same. The decay curve of the product sepd. by (2) shows a greater initial drop which is probably due to a product which he calls U- $\gamma$  having a half period of 1.5 days. It could not be completely sepd. from U-X. It emits  $\beta$  rays and  $\alpha$  rays, the presence of latter being detd. by measuring the rate of scintillations over a 3-day period; the half-period value was 1.5 days. U- $\gamma$  is not an intermediate product between U and U-X but a lateral disintegration product. The quantity is small compared with U-X.

The Nature of Gamma Rays. T. H. LABY AND P. BURBIDGE. Physic. Lab., Victoria Coll., Univ. N. Z. Nature, 87, 144.—Some expts. are described which show that fluctuations occur in the ionization produced by  $\gamma$ -rays in the same way that ionization by  $\alpha$  rays, which is due to a finite number of independent events, is subject to fluctuations. This result would be explained if (1) the  $\gamma$ -rays from Ra are projected particles, or (2) if the number of ions produced in air by a const. source of  $\gamma$ -rays is subject to fluctuations. The expts. are being conducted with a view to detg. what part each of these factors play in producing the fluctuations observed.

W. H. Ross.

Transformation of the Energy of Homogeneous Röntgen Radiation into Energy of Corpuscular Radiation. Charles A. Sadler. Phil. Mag., 22, 447-58.—Primary X rays from a focus tube impinged on a plate of metal which in turn became a source of homogeneous secondary rays. Some of these entered an ionization chamber at the bottom of which a tertiary radiator was placed. The total ionization produced in the chamber is then, that due to the secondary radiations absorbed while traversing the air of the ionization chamber plus the tertiary radiations produced by the plate at the bottom of the chamber plus the ionization produced by corpuscles projected from the tertiary radiation. The tertiary radiator became the source of a corpuscular radiation which is absorbed by a few mm. of air. Several elements were used as secondary radiators to obtain a range in penetrating power of the homogeneous secondary rays. A large number of tertiary radiators were used. The expts. confirm the theory that Röntgen radiation is always accompanied by the emission of negative corpuscles and that whenever a radiation characteristic of an element is excited there is always produced a strictly proportional amt. of corpuscular radiation. R. F. E.

Spectra of Fluorescent Röntgen Radiation. CHARLES G. BARKLA. Phil. Mag., 22, 396-412.—A review and discussion of a series of disconnected articles on the general subject of homogeneous secondary radiation which have been published by the author and by Barkla and Sadler.

R. F. E.

The Anti-cathodes of X-Ray Tubes. J. Schofield. Nature, 87, 215.—An X-ray tube fitted with an anti-cathode of carborundum was found to work very successfully, and did not seem to pass so readily into the hard condition as tubes fitted with the more expensive materials as Pt, Ta, etc.

W. H. Ross.

The Alpha Theory of Aurora Borealis. L. Vegard. Univ. Christiana. Nature, 87, 213-4.—A number of arguments are given in addition to those already announced (C. A., 5, 2368) in support of the view that the properties of auroral drapery bands, and probably other forms of aurora, are best explained by assuming that they are caused by  $\alpha$  rays from some radioactive substances on the sun. According to this theory each of the drapery bands must be produced by a homogeneous  $\alpha$  radiation, and such a radiation is known to be given out during one radiaoctive transformation, while the homogeneous groups given off from radioactive compds. formed through the process of at. disintegration may be assumed to give rise in the same way to the peculiar prop-

erty of the aurora in forming parallel bands. If this is the case then the auroral drapery bands should form a kind of magnetic spectrum of the  $\alpha$  rays given out by the radioactive matter present in the sun, which would thus open up the possibility of studying and identifying the radioactive substances on the sun by examining the magnetic  $\alpha$  ray spectrum produced by the earth's magnetic field in the form of drapery bands.

W. H. Ross.

## 4. ELECTROCHEMISTRY.

C. G. FINK.

Water Power Development on the Mississippi River at Keokuk, Iowa. Anon. Eng. News, 66, 355-65.

J. J. M.

Aluminium versus Copper. C. H. PALMER. Elec. Rev. West. Elec., 59, 523.—
The author cites a number of 3,000 v. transmission lines using Al in place of Cu. Hard drawn Al and Cu wires will have the same cond. if the former has 1.64 times the sectional area and consequently 1.28 times the diam. of the latter. By the use of Al 25% on the total cost of a transmission line can be saved. D. K. WRIGHT.

Resistance Material with Variable Temperature Coefficients. H. KÖRBER. Z. angew. Chem., 24, 1402-5.—With the exception of manganin, all metallic resistances have positive temp. coeffs. The metallic-ceramic resistance material of Parvillié, which consists of powdered Ni, SiO<sub>3</sub>, kaolin and certain fluxes to minimize oxidation of the Ni, fused together and hydraulically compressed into rods or plates is very efficient when new but the metal oxidizes after a relatively short time, the portions near the terminals fuse and the resistance fails. The author attributes this to the base metal and therefore employs as a successful substitute a noble metal, preferably Ag (C. A., 5, 1234). The addition of Al or Mg is absolutely necessary to insure uniform distribution of the Ag and to prevent its fusion. The Al<sub>2</sub>O<sub>3</sub> or MgO formed, acts as an additional resistance and dets. the sp. resistance of the substance. There is no fusion in the chem. process during firing. The resistances have a high current capacity, a rod 100 mm. long and 3-4 mm. sq. section carries 2 amp. Data illustrating the actual use of the material is appended.

J. S. GOLDBAUM.

Resistance Materials for Electrical Furnaces. F. Bolling. Elektrochem. Z., 17, 331-3.—The author discusses the comp. of silundum, maintaining that it is SiC and therefore carborundum and not the product which Acheson discovered and called "siloxicon." A furnace made by A. Sieverts is described. It consisted of a silundum tube 40 cm. long, 4 mm. thick and 48 mm. inside diam. The furnace was used up to 1700° without difficulty even though this is somewhat above the ignition point of the silundum. At temps. over 1500°, SiO, vapors filled the tube. A tube made of ground carborundum and a binding agent is also used by Sieverts but, it is contended by the author, this is neither as cheap or as satisfactory as the regular silundum.

W. E. RUDER.

Electric Tube Furnaces with Calorite Resistors for the Laboratory. S. A. Tucker. J. Ind. Eng. Chem., 3, 595-8.—A detailed description with sketches of a porcelain tube furnace wound with "calorite" ribbon, and successively insulated by alumina, asbestos tubing, asbestos wool, and an asbestos container. Two types, a horizontal tube, and a crucible type are shown, and sketches given of tubes with various spacings of the winding for different temps.

W. L. VAN KEUREN.

The Trollhittan Electric Smeltery. J. B. Van Brussel. Eng. Mining J., 92, 650-2; see Leffler, C. A., 5, 3197 and following abstr. ROBERT KANN.

The Electric Pig Iron and Steel Plant at Trollhättan, Sweden. J. A. LEFFLER. Engineering, 92, 374-9; Met. Chem. Eng., 9, 505-10; cf. C. A., 5, 3197. J. J. M.

Transforming Stations of Niagara Electrochemical and Electrometallurgical Industries. A. J. Jones. J. Ind. Eng. Chem., 3, 756-70.

J. J. M.

A Combined Besseme: and Electric Furnace. Anon. Elec. Rev. West. Elec., 59, 648.—The app. consists of a double-ended converter, which can be tilted through an angle of 180° or more. One end is provided with tuyers for blowing, and at the other end are the electrodes, with cooling jackets of cold air or water. The Bessemer part of the furnace is run as in the ordinary Bessemer converter, when this is completed, the furnace is tilted and the metal transferred to the portion which contains the electrodes. Refining can here be carried to any desired extent, both arc and resistance heating being obtained in the furnace. For complete removal of S and P about 190 kw. hours are required.

G. F. YESSLEER.

Experiences with the 15-Ton Héroult Furnace at the South Works of the Illinois Steel Co., South Chicago. C. G. OSBORNE Chem. Eng., 13, 118-22; cf. C. A., 4, 2413.—A detailed description of the construction, operation, and variety of products of the furnace are given. The main chem. and physical differences in elec. steel as produced are mentioned as; (1) comparative freedom from oxidation; (2) comparative freedom from segregation; (3) higher tensil strength and slightly higher ductility for the same chem. analysis; and (4) a steel of greater d. than other com. steels with the possible exception of crucible steel.

C. N. Moore.

Tool Steel Direct from the Ore in an Electric Furnace. A. STANSFIELD. J. Canadian Min. Inst., 13, 151-62; J. Soc. Chem. Ind., 30, 286.—Expts. were made upon the direct production of steel from magnetite ores containing Ti and traces of V. The furnace used was cylindrical, 18 in. high, and 14 in. in diam., with 2 lateral, graphite electrodes 1 in. sq. 200 amps. at 110 v. was the max. power used. The slag and ore were first fused by an arc between electrodes and then with electrodes dipped in the bath. The ore used contained 51.45% Fe, 7.5% Ti, 0.12% Ni, and traces of V and S. The charge consisted of 100 parts ore, 20 CaCO<sub>3</sub> and 18 of C, formed into briquets with molasses and water (10-16). No addition of Fe-Mn, or other deoxidizing agent was used before tapping, and sound ingots (3 lbs.) were invariably obtained. This was not possible with non-titaniferous ore. This product proved to be good quality tool steel and the total cost of ore, C, CaCO<sub>3</sub>, power, and electrodes is estimated at 2 cents per lb. of steel for this small furnace.

W. E. Ruder.

Steel Castings from Electric Furnace. C. H. Von Baur. Chem. Eng., 14, 332.—
The furnaces are of the Röchling-Rodenhauser induction type, operating on single, 2 or 3-phase a. c. (25 cycle preferred). The furnace action is similar to any ordinary transformer. The metallurgical course of the process is similar to the one employed in the basic open hearth furnace. Elec. steel can easily be made to meet strict specifications. Steel can be melted in the elec. furnace for \$4.20-3.50 per ton, depending on the size of the furnace, with electricity at 0.6 cents per kw. hr.

G. F. YESSLER.

The Electrometallurgical Revolution in the Iron and Steel Industry of Norway and Sweden. J. W. RICHARDS. Proc. Eng. Soc. Western Penn., 27, 125.—Several well-known types of elec. steel furnaces are described and illustrated. The operation of the pig-iron furnace is explained, and the relationship shown between amt. of fuel, CO<sub>2</sub> and CO in the gas, and efficiency.

HORACE C. PORTER.

Electric Tin Smelting. JOHN HÄRDÉN. Met. Chem. Eng., 9, 453-7.—Advantages of elec. furnaces over reverberatory furnaces are: loss by volatilization reduced; formation of "hardhead" minimized; a steady production of metal; amt. of C necessary is only 17% instead of 25; less labor; less space. The most economical method is to

produce first a slag containing 14-16% of Sn and to treat this richer slag, while still hot, in a second furnace. The reaction is run on 50% of each equation:  $SnO_2 + C = Sn + CO_2$ ;  $SnO_2 + 2C = Sn + 2CO$ . Theoretically one ton of Sn should be produced on 1140 kw. hrs. while the best practical result was 1700 kw. hrs. per t. A test was run on a furnace resembling the Harmet for Fe ore reduction. It was provided with a bottom chamber of magnesite bricks, rammed with calcined magnesite and tar. A charging shaft rested on a vault or arch. Two lateral and inclined C rods, about  $2^{1}/_{2}$  in. in diam. were used as electrodes and reached into the center of the hearth about  $2^{1}/_{2}$  in. from the bottom. 16 lbs. of ore were smelted, giving a total of 9 lbs. of metal which was 90% of the metal in ore. The max. power was 8.4 kw. and the time for melting 2 hrs. The total power used was about 4900 kw. hrs. per ton of metal. The tin obtained direct from the furnace was over 99% pure; it contained very little Fe and no As. (Also in *Electrician*, 67, 964.)

G. F. Yessler.

Experiments on the Electrothermic Treatment of Zinc Minerals. F. Sartori. Monteponi. Resocon. rass. min. Sarda, 15, No. 5; Rass min., 35, 3-4.—The results of expts. carried out with different types of elec. furnaces are not yet of such a nature as to make it possible to decide whether such processes can be made practicable. With one furnace which is yet under investigation and in which the charge (consisting of calcined calamine and coke or coal in excess) is not fused (patented by the Soc. Monteponi), the best yield so far has been 2.70 kg. Zn per kw.-day.

Chas. A. Rouiller.

Silent Electric Discharges in Gases at Atmospheric Pressures. E. H. RIESEN-FELD. Stockholm. Z. Elektrochem., 17, 725-31.—With the aid of a Januasch ozone generator (J. prakt. Chem., 73, 499 (1906)) the minimum potentials necessary to effect a discharge through the gas, and the continuous potentials, i. e., the pot. for an uninterrupted discharge, were detd. for all of the common gases at atm. pressure. The values for the minimum pot. are proportional to those for the cont. pot. The relative min. pot., i. e., the pot. based on that of O taken as unity, is well defined for each gas, and independent of the shape and size of the ozonator tube, the rate of flow of gas through the tube, and the frequency of the current (a. c.). The values obtained by gradually increasing the pot. are identical with those obtained by starting with a high pot. and gradually decreasing it. Small amts. (1%) of impurities do not effect the readings noticeably. The higher the current frequency, the higher both the min. and cont. pot. values. Irregularities were met with in the case of SO, and Cl. The relative min. pot. values are: H, 0.77; NO, 0.85; CH, 0.97; NH, 0.97; O, 1.00; CO, 1.03; CO, 1.032; C,H, 1.11; N, 1.16; H,S, 1.27; HCl, 1.29; Cl, 2.41; SO, above 3.0. The glow during discharge takes on a tint distinct for each individual gas. There does not seem to be any close relation between silent discharge and spark phenomena. Gases with high diffusion coeffs. have low min. potentials. The greater the tendency of a gas to form negative electrons the lower will be its min. pot.

Electric Discharges in Gases at Reduced Pressures. L. A. PARSONS. Trans. Am. Electrochem. Soc., 19, 109-18.—A review of the progress of the last 5 years.

L. K. MULLER.

Warm and Cold Gold Baths. G. Nicolaus. Elektrochem. Z., 18, 145.—Heated baths for Au plating give a warmer tone to the deposit and a mat surface for antique finishes. The bath should not be allowed to get above 75° and a lower c. d. is used than with the cold soln. Small articles which are to have only a Au coloring are plated in a warm bath. For larger articles cold baths are used. Formulas and methods of prep. are given for both hot and cold electroplating. Formulas for gilding by dipping are also given.

W. E. Ruder,

Electrolytic Production of Oxygen and Hydrogen; and Welding and Cutting by the

Oxyhydrogen Flame. Anon. Met. Chem. Eng., 9, 471-3.—The cell consists of an Fe tank for the cathode; an Fe cover from which is suspended a hydraulic joint and a perforated tank for the anode. An asbestos sack is suspended from the hydraulic joint forming 2 sep. compartments. Through an opening in the cover a soln. of caustic alkali in distilled water is poured into the hydraulic joint and distributed in the 2 compartments. Each unit cell requires a little over 2 v. and 300-400 amp. A current of 350 amp. produces 65 cu. ft. of O and 130 cu. ft. of H per day. The oxyacetylene flame is specially suitable for welding heavy pieces, while the oxyhydrogen flame is far superior for thin sheets. In oxy-cutting, O and C<sub>2</sub>H<sub>2</sub> will cut 4-6 in., whereas with O and H cuts up to 24 in. have been obtained. The purity of the gas is an important factor.

G. F. Yessler.

Treatment of the Anode Sludge in Electrolytic Refineries. E. F. KERN. Met. Chem. Eng., 9, 417.—Anode sludge consists mainly of the associated impurities which stand lower in the e. m. f. series than the metal which is being refined. Analyses of Cu anode sludge (in %) follow: 15.0-55.0 Cu; 2.0-8.0 Sb; 1.5-6.0 As; 0.2-8.0 Bi; 0.5-6.0 Pb; 0.5-12.0 S; 0.5-1.5 Fe; 0.1-2.5 Se; 0.1-3.5 Te; 5.0-50.0 Ag; 0.017-0.70 Au, Analyses of Pb anode sludge (in %) were as follows: 6-18 Pb; 5-25 Cu; 5-70 Sb; 2-50 As; 0-20 Bi; 0-2 Sn; 0-3 Se; 0-4 Te; 10-40 Ag; 0-1 Au. A characteristic of Pb anode sludge is that the constituents are present principally as metals or oxides, whereas the constituents of Cu anode sludge are present principally as basic sulfates, sulfates and oxides. The sludge is screened, washed and dried and then treated for metal content by one of the following methods: (1) Direct cupellation, (2) fusion method, (3) fractional scorification in basic lined reverberatory furnaces, (4) direct parting with b. conc. H<sub>2</sub>SO<sub>4</sub>, (5) mixing the washed and dried sludge with a calc. amt. of conc. H<sub>2</sub>SO<sub>4</sub>, (6) treatment with hot dil. H,SO4, air and KNO4. The article discusses these methods in G. F. YESSLER. detail.

A New Primary Cell. STEPHAN BENKÖ. Met. Chem. Eng., 9, 401.—B. has improved the mechanical construction on the ordinary Zn-Na dichromate cell so that polarization at the C electrode is overcome. It consists of a flattened C cylinder open at both ends. The C is provided with a Pb cap at the bottom and a Pb ring at the top, which carries one terminal, thus providing a vessel open only at the top. A shell of sheet Pb is joined to the cap at the bottom and the ring at the top so as to form a chamber all around the C. The Zn is inserted into the inner chamber and carries the second terminal. The electrolyte in the outer chamber percolates through the C to the Zn and flows off through a Pb tube, fitted to the Pb base, maintaining the level constant within the C. The flow of electrolyte can be so arranged as to maintain a constant current. Two electrolytes are recommended—for electric light, 60 g. of Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> and 120 cc. H<sub>2</sub>SO<sub>4</sub> per l. of H<sub>2</sub>O; and for heavy currents, 100 g. of Na<sub>2</sub>Cr<sub>2</sub>O, and 150 cc. H<sub>2</sub>SO<sub>4</sub> per 1. of H<sub>2</sub>O. The internal resistance is below 0.01 ohm. There is also some polarization but it is difficult to sep. it from the internal resistance. The cost of operation is about 50 cents per kw. hr. G. F. YESSLER.

Monochromatic Light and Visual Acuity. M. Luckiesh. Elec. World, 58, 450.

—A full account of expts. showing the superior defining power of monochromatic light. By the use of absorbing solns. and glasses, lights of the same hue but of different spectral character are produced. In nearly every case observed the more nearly monochromatic source required less illumination for the same distinctness of detail.

W. L. VAN KEUREN.

The Primary Standard of Light. EDIT. Elec. World, 58, 427.—Taking into account all the essential relationships between light and energy, the following standards are proposed. C. P. Steinmetz standard is based on the Hg vapor lamp as the source, selecting 3 monochromatic radiations in specified proportions. H. E. Ives suggests

the monochromatic green Hg radiation which is of sufficient intensity to measure, both as radiation and as light. Houstoun adds to Ives' suggestion, the idea of measuring the radiation in abs. units ( $x = \frac{x}{2}$  ec.) through an ideal screen. In the event of these suggestions becoming practicable, the units of flux per watt of any illuminant will bear a definit relation to the efficiency.

W. L. Van Keuren.

Cooper-Hewitt Silica Lamps. Anon. Elec. Rev. West. Elec., 59, 540.—A new type of silica lamp has recently been put upon the market. It operates on d. c. 1.5 amp., 200–250 v. and gives 800 c. p. The lamp is started by automatic tilting. At first the color is blue in a very short time becomes almost white, owing to the high temp. of operation. It will stand 10% voltage fluctuations above or below normal without trouble. The av. life per burner (SiO<sub>2</sub> tube) is 2000–3000 hrs. W. E. RUDER.

Observations on the Effect of the Light of the Mercury Vapor Lamp on the Bys. C. H. WILLIAMS. Elec. World, 58, 550-2.—Thorough ophthalmological exam. of persons who had regularly, and for long periods, worked by the light of the Hg are showed quite as good conditions as would be expected in any other similar group working much by artificial light. Temporary color fatigue was clearly observable immediately after leaving work.

C. N. MOORE,

Physiological Effects of Mercury Arc. Edit. Elec. World, 58, 530-1.—Comment upon William's article (cf. abstract above).

C. N. Moore.

The Osram Lamp and its Applications. H. Remans. Berlin. Electrician, 67, 829.—The special advantages which the "Osram" (W) lamp presents, especially in street lighting, are fully discussed.

G. F. Yessler.

Four Years of the Tungsten Light. EDIT. Elec. World, 58, 527.

W. L. VAN KEUREN.

Fusing Wires in Hydrogen. A. A. SOMERVILLE. Elec. Rev. West. Elec., 59, 583-4.—A description of a method of fusing Cu wires onto some other metal which has a high m. p. and oxidizes readily. A typical case is that of fastening Cu leads to W, Mo or Ta filaments for incandescent lamps. The app. consists of a tall quart bottle of dark-colored glass with the bottom removed. The bottle is filled (through the neck) with H or a mixt. of H and N. The tips of 2 pointed C electrodes are brought together and a small arc is made to play on the tips of the filament and wire to be fused together until a bright globule of molten metal is formed.

D. K. WRIGHT.

The Theory and Construction of Mercury Watt Meters. W. KESSELDORFER. Elektrotechn. Z., 32, 684.—A historical and theoretical consideration of all types of meters using Hg cups or contacts and a detailed description of their application in kw. hr. meters.

W. E. RUDER.

Dynamo Brushes. Anon. Elektrochem. Z., 18, 147.—A new substitute for brushes of Cu, brass or bronze consists of Cu 98%, Bi 0.2%, Cd 0.3% and Sb 1.5%. The mixt. is made by heating or by mixing and pressing the powdered constituents. The brittle, cryst. structure of this combination is claimed to have marked advantages for commutation.

W. E. RUDER.

Production of Metallic Coatings by Friction. O. HILDEBRAND. Elektrockem. Z., 18, 143.—The theory of the process is reviewed and directions given for Cu plating, Zn, Sn, Fe, steel, and cast Fe. For plating on Zn a mixt. of CuO, tartaric acid and chalk is used. The same mixt. plates well on Fe, steel and Sn if powdered Zn is used with it. Cast Fe, wrought Fe, and steel are best coated with a mixt. of 66 pts. of powdered CuSO<sub>4</sub> and 34 of tartaric acid. Acid CuSO<sub>4</sub> soln. is recommended for small Fe and steel articles.

W. E. RUDER.

Asbestos. J. F. SPRINGER. Iron Age, 86, 1487.—A short review of its occurrence and methods of manuf.

L. A. TOUZALIN.

Air Nitrogen Fertilizers (MILO). 28.

Transformer Oil (DUCKHAM). 22.

Ultraviolet Radiation of Vapor Lamps (HENRI). 3.

- U. S., 999,749, Aug. 8. L. W. Chubb, Wilkinsburg, Pa. Assignor to Westinghouse Elec. and Mfg. Co. Preparing an edgewize-wound aluminium strap coil for electric arc lamps, dynamo field magnet windings, etc., by bending the Al strap into an open helix, immersing in a bath of Na silicate, passing an elec. current through the helix as one electrode and the bath as the other to form an insulating film on the Al and finally pressing the adjacent convolutions of the helix together.
- U. S., 999,762, Aug. 8. T. A. Edison, Llewellyn Park, Orange, N. J. Assignor to Edison Storage Battery Co., West Orange, N. J. Active material for the negative electrodes of storage batteries, comprizing electrolytically active, finely divided Fe and Bi amalgam.
- U. S., 1,000,330, Aug. 8. N. Dodge, Philadelphia, Pa. Assignor to Elec. Storage Battery Co., same place. Storage battery separator consisting of a sheet of fine grain wood having its normal structure and containing its cellulose and fibrous constituents and constituting a diaphragm impervious to battery sediment and deprived of such of its constituents as would deleteriously attack Pb when subjected to electrolytic action and retaining such of its constituents as beneficially effect a negative plate; the wood being prepared for use by soaking successively in dil. H<sub>2</sub>SO<sub>4</sub> and KOH solns.
- U. S., 1,000,717, Aug. 15. W. T. CONN, Lakewood, Ohio. Assignor to National Carbon Co., Cleveland, Ohio. Electrode for flaming arc lamps, having an Al resistance reducing wire extending lengthwize therethrough and enveloped by the material of the electrode. The Al wire may be electroplated with Cu to prevent oxidation.
- U. S., 1,000,761, Aug. 15. J. L. K. SNYDER, Cleveland, Ohio. Assignor to National Carbon Co., Cleveland, Ohio. Applying a protective coating to an electric furnace carbon electrode, by applying to the surface of a green unbaked C electrode a paste comprizing a binder such as water glass, pitch, tar, glue or glucose and a chemical, e. g., CaCl<sub>2</sub>, Na<sub>2</sub>CO<sub>2</sub>, KHSO<sub>4</sub> or borax, which under the influence of the curing heat will produce a slag-forming protective coating on the surface of the electrode, and finally heating the paste-covered electrode to a curing temp.
- U. S., 1,000,805, Aug. 15. A. HELFENSTEIN, Vienna, Austria-Hungary. Electric furnace having a pair of girders extending from end to end of the reduction chamber at its top and spaced apart so as to form a charge opening extending the entire length of the chamber, a receptacle above the girders and supported by them to confine the charge, a vertical longitudinally placed electrode between the girders and extending nearly the entire length of the furnace, and an electrode arranged in the bottom of the furnace.
- U. S., 1,000,838, Aug. 15. H. NATHUSIUS, Friedenshütte, near Morgenroth, Ger. Electric furnace with a group of upper electrodes and a group of lower electrodes, a poly-phase a. c. generator and circuit-forming connections therefrom to the electrodes so that an a. c. may be passed through the material in the furnace, a substantially neutral zone of the a. c. circuit being maintained in the bath itself.

### 5. PHOTOGRAPHY.

#### LOUIS DERR.

Simultaneous Exposure and Development. O. MENTE. Atelier Phot., 18, 66-9.

—A particularly successful process for copying hard negatives on bromide paper. The paper is wetted with 10 cc. rodinal, 20 cc. glycerol, 40 cc. water, and exposed in the copying camera while wet, for a time long enough to print the shadows of the picture. An orange glass is then placed before the lens, and this, with a sheet of ground glass to diffuse the light, enables the development to be watched. When the shadows are sufficiently dense the glasses are removed and an additional exposure given for the high lights; but this does not over-expose the shadows, because the already developed Ag image there screens the AgBr beneath.

L. Derr.

Honeycomb Markings on Negatives. H. LÜPPO-CRAMER. Phot. Industrie, 1911, 995.—These are produced by development without rocking the tray. The author obtained similar appearances by atmospheric oxidation of simple solns. of hydroquinone and pyro in shallow trays, and concludes that they are produced only with readily oxidizable developers containing relatively little sulfite.

L. Derr.

The Characteristic of the Amidol Developer. E. FLORENCE. Phot. Chronik, 18, 252-4.—In simple soln. it has no developing power because of its HCl content. Addition of enough Na<sub>2</sub>SO<sub>3</sub> to sat. the HCl liberates H<sub>2</sub>SO<sub>4</sub> and confers a slight developing power. Enough Na<sub>2</sub>SO<sub>3</sub> to convert the acid into NaHSO<sub>3</sub> increases the developing power greatly, the best result being obtained with 1 part amidol to 6 parts sulfite. The acid reaction of the developer can be strengthened by adding C<sub>2</sub>H<sub>3</sub>O<sub>7</sub>, but the developing power is not affected thereby except for large additions.

L. Derr.

Intensification in One Bath. F. GOUILLON. Photo-Revue, 1911, 146.—Take PtCl<sub>2</sub> 1.5 g., Hg<sub>2</sub>Cl<sub>2</sub> 1 g., water 600 cc. Plates completely free from hypo are immersed in this bath until sufficiently intensified, and are then washed for a short time. The given bath keeps well, and is enough for 10  $5 \times 7$  plates.

L. Derr.

The Silver Pigment Process. J. Stoop. Atelier Phot., 18, 90-6.—Detailed working directions for the ozobrome process.

L. D.

New Applications of Artigue Paper or Gum Prints. C. Gravier. Bull. soc. franç. phot., [3] 2, 242-3.—Pour 6% collodion on the print. After this dries, the print is immersed in cold water and the collodion side pressed on glass; the water is then poured off and boiling water applied for 15 sec., whereupon the paper becomes detachable from the image, which can be transferred to another support. This is applicable to engravers' uses.

L. Deer.

Improvements in Methods of Developing after Fixing. A. AND L. LUMIRE AND A. SEVEWETZ. Bull. soc. franc. phot., [3] 2; 264-9.—A 2% hypo bath is the best fixing agent for studies of this kind, the ordinary bath partially destroying the image; the dilute bath allows a decided reduction in the exposure necessary to give good images. As developers, AgNaSO<sub>2</sub> and HgNaSO<sub>3</sub> were found best.

L. Derr.

Microscopic Observations on Ripening. H. LÜPPO-CRAMER. Phot. Rundschaus, 25, 176-7; 9 photomicrographs.—Ripening is always accompanied by an increase in the size of grain, from the ultramicroscopic dimensions of the colloidal AgBr on a Lippmann plate up to 5-10  $\mu$  of the rapid ordinary plate, and all the intermediate stages can be followed in the microscope. Ripening being effected by NH<sub>2</sub>, the author coated a plate with colloidal AgBr emulsion and exposed it to NH<sub>2</sub> vapors for 1 hr. after drying. The enlarged grains are plainly visible in the microscope. Ordinary dry plates similarly treated show like results, the larger AgBr complexes growing at the

expense of the surrounding areas, and each grain being bordered by a perfectly clear area from which the AgBr has been abstracted by the central nucleus. L. Derr.

Physical Development with Printing-out Papers. R. Beckers. Phot. Rundschau, 25, 186-9.—Printing is carried to visibility of all details. The image is then developed to the desired density in a hydroquinone, pyro, or metol developer containing Na<sub>2</sub>SO<sub>3</sub> and C<sub>2</sub>H<sub>2</sub>O<sub>7</sub> but no alkali, and then is fixed. With K<sub>2</sub>CrO<sub>7</sub> and C<sub>2</sub>H<sub>2</sub>O<sub>7</sub> and alcoholic pyro applied in varying proportions after fixing, green, blue, brown and red tones can be obtained.

L. Derr.

Differentiation, by Development, between the Latent Images on Silver Chlorids and Bromide Gelatin Emulsions. A. AND L. LUMIRRE AND A. SEYEWETZ. Bull. soc. franc. phot., [3] 2, 168-71.—Developers whose developing power lies wholly in the OH groups will develop latent AgCl images without markedly affecting images on AgBr. Na quinonesulfate, in presence of Na<sub>2</sub>SO<sub>3</sub>, possesses this power in a marked degree, and may be utilized to detect the presence of AgCl in emulsions of other Ag halides.

L. Derr.

## 6. INORGANIC CHEMISTRY.

#### H. I. SCHLESINGER.

The Sulfides of the Earth Metals. W. BILTZ. Clausthal. Elektrochem. Z., 17, 668; Z. anorg. Chem., 71, 427-38.—Al<sub>2</sub>S<sub>2</sub> was prepared according to the thermite process from a powdered mixt. of Al and S. The raw product contained 2-5% AlS and was purified by sublimation in vacuo. M. p. = 1100  $\pm$  10°; hexagonal needles; very hygroscopic. CeS<sub>2</sub> decomposed at 790°; LaS<sub>2</sub> at 670°. LaS<sub>3</sub> hydrolyzed readily. Boron sulfide melted at 310°, and sublimed easily. The La sulfides could not be sublimed nor melted in vacuum at 1250°. C. G. FINK.

New Determinations of the Ratio of the Molecular Weights of Potassium Chiorate and Potassium Chloride. ARTHUR STÄHLER AND FRIEDRICH MEYER. Chem. Inst., Univ. Berlin. Z. anorg. Chem., 71, 368-402.—The ratio KClO./KCl was detd. by decomposing a known amt. of KClO, by evapn. with HCl, and weighing the KCl formed. KClO, was prepd. by recrystn. in Pt first from H<sub>2</sub>O to which a little NaOH had been added, then from pure H<sub>2</sub>O. The purity of the KClO<sub>2</sub> was checked by detg. the Cl content with the nephelometer. The m. p. was detd. as 357°; d18, 2.337. KClO2, contrary to the statements of Stas, cannot be fused without decomp. Pure KClO<sub>2</sub>, dried in a desiccator over KOH was weighed in a tared quartz flask, after being brought to const. wt. at room temp. by a current of dry air, and fused in a current of dry air, collecting the H<sub>2</sub>O evolved in P<sub>2</sub>O<sub>4</sub>. The wt. of H<sub>2</sub>O-free KClO<sub>2</sub> was then detd. by difference. H<sub>2</sub>O was then added to the fused KClO<sub>2</sub>, and the latter reduced by means of a current of HCl. In order to completely retain particles mechanically carried over, the Cl and Cl oxides evolved were condensed by a mixture of EtOH and liquid air, at -50 to -60° and the HCl by liquid air. After decomp, was complete the contents of the decomp. flask were evapd. to dryness, dried, fused and weighed. The condensed gases were allowed to evap. slowly, and the solid, consisting mainly of KClO4, treated with HClO4, weighed as KClO4 and the detn. corrected. The mean value for the ratio KClO<sub>2</sub>/KCl obtained in 5 detns. comprizing the final series is 1.643819. Assuming 0 = 16.000 and the ratio K/Cl = 1.102641 (Richards and Stähler) the at. wt. of  $K = 39.097 \pm 0.0004$ , that of  $Cl = 35.458 \pm 0.0004$ .

GEORGE W. MOREY.

Preparation of Barium Nitrate. I. Equilibrium in the Ternary System: Barium Mitrate, Sodium Nitrate, Water. ANGELO COPPADORO. R. ist. tech Asti. Rass. min.,.

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35, 121-4.—Detns. of the solubility at 0° of Ba(NO<sub>2</sub>), and NaNO<sub>2</sub> in the presence of each other and of the comp. of the solid phases by the Schreinemaker method show that the 2 compds. form no double salt, there being but a single satd. soln. containing 1.55% of the Ba salt and 41.30% of the Na salt.

Chas. A. Rouiller.

Destruction of Traces of Iodine. Action of Mixtures of Hydrogen Peroxide and Ammonia on Iodine. L. FOURTON. San Jacinto, Mexico. Mem. rev. soc. cien. "Antonio Alzate," 28, 275-84.—I in aq. soln. is rapidly destroyed on adding a mixt. of H<sub>2</sub>O<sub>2</sub> and NH<sub>3</sub> water. Detn. of the ratios I/NH<sub>3</sub> and I/O for I and NH<sub>4</sub> consumed and O evolved (on adding H<sub>2</sub>O<sub>2</sub> and NH<sub>2</sub> water in various proportions) indicates that the principal reaction is approx. that represented by the equation: I, + 2NH, +  $H_2O_3 \longrightarrow 2NH_2I + O_3$ . The ratio I/NH, is constant but variations in the ratio I/O indicate that one or more secondary reactions are superimposed on the above. Part of this additional evolution of O may be accounted for by the reaction between NH, and H<sub>0</sub>O, and possibly by a reaction like  $2NH_1 + I_2 + H_2O \longrightarrow 2NH_2I + O$ in which the ratio I/NH, is the same as in the equation above. Destruction of I by NH, and H,O, likewise occurs when the I is dissolved in alc, or in KI soln, or when the hydroxides or carbonates of K and Na are substituted for NH<sub>2</sub>. The action of H<sub>2</sub>O<sub>2</sub> and NH<sub>2</sub> on I may be utilized in a practical way for removing I stains from linen cloth, etc. For this purpose use 1 pt. com. NH<sub>2</sub> water and 3 pts. com. H<sub>2</sub>O<sub>2</sub>, then wash out with H<sub>2</sub>O. H. S. PAINE.

Nature of the Photoelectric Property of Selenium. G. Gallerani. Phys. Physiol. Inst. Camerino. Boll. soc. Eustach., 8, 1-15; through Zentr. Biochem. Biophys., 11, 865.—Theoretical.

I. K. Phelps.

Nomenclature of Hydrogen Peroxide Derivatives. A. v. ANTROPOFF. Chem. Ztg., 35, 670.—By the addition of O to chlorides, sulfides, etc., chlorates, sulfates, etc., resp., are obtained; likewise in the case of oxides the metallic compds. containing more O (derivs. of  $H_2O_2$ ), i. e., salts of  $H_2O_2$  should not be called peroxides, superoxides or peroxidates but oxates. The Hg salts of  $H_2O_2$  may be called mercurous oxate ( $Hg_2O_2$ ) and mercuric oxate ( $HgO_2$ ). The acid Na salt of  $H_2O_2$ , called Na hydroperoxide by Baeyer, may be called Na hydroxate. Such a nomenclature would avoid confusion with true superoxides.  $H_2O_2$  itself would be hydrogen oxate or hydroxate or in case its acid character is to be emphasized hydroxatic acid. This confusion in nomenclature arising from the multivalence of metals does not occur in organic chemistry, but since Me peroxide is the  $H_2O_2$  ester of MeOH it could, analogously with Me sulfate, be called methyl oxate.

E. J. WITZEMANN.

Nomenclature of Hydrogen Peroxide Derivatives. E. EBLER. Heidelberg. Chem. Zig., 35, 724.—An article by Antroposi (see preceding abstr.) led E. to call attention

to one by him (C. A., 5, 3019) in which he proposes to call salts of the type  $M \subset \prod_{i=0}^{|I|}$ 

"peroxites" in distinction from peroxides of the type M "Peroxidate" (Bredig

and V. Antropoff, Z. Elektrochem., 12, 588), he applies to addition compds., as KF.H<sub>2</sub>O<sub>3</sub> (Tanatar, Z. anorg. Chem., 28, 255) in analogy to hydrate, ammoniate, hydrazinate, hydroxylaminate, etherate, aminate, etc., for addition compds. of H<sub>2</sub>O, NH<sub>2</sub>, N<sub>2</sub>H<sub>4</sub>, etc. The name "peroxite" is analogous to hydroxylamite, previously suggested (J. prakt. Chem., 78, 327) for the true salts of hydroxylamine. D. M. LICHTY.

Coefficient of Magnetism of Gold (HANRIOT, RAOULT). 2.

# 7. ANALYTICAL CHEMISTRY.

#### E. G. R. ARDAGH.

Review of the Work in Analytical Chemistry during 1910. M. HUYBRECHTS.

Bull. soc. chim. belg., 25, 328-52.

I. K. PHELPS.

'Tin and its Methods of Assay. José Zárate. Mexico. Mem. rev. soc. cies. "Antonio Alzate," 28, 193-7.—Discussion of a modification of the method of Pearce and Low for the rapid volumetric estn. of Sn. H. S. Paine.

The Volumetric Determination of Ferric Iron with Permanganate after Reduction with Zinc. E. MÜLLER AND G. WEGELIN. Stuttgart. Z. anal. Chem., 50, 615-23.

—The reduction of ferric solns. with pure Zn is fairly rapid at 100°, but unless all the Zn is finally dissolved, the results are not accurate, as Fe is deposited on the Zn. Nothing is gained by having Pt in contact with the Zn. No Fe is deposited on amalgamated Zn, and as less Zn is dissolved the error due to any impurities in the Zn is less. The addition of a small amt. of Cu accelerates the reducing action of the amalgamated Zn. Percy H. Walker.

Farric Sulfate as a Standard for Permanganate Titration. J. MILBAUER AND O. QUADRAT. Bohem. Tech. High Sch., Prag. Z. anal. Chem., 50, 601-3.—Fe<sub>3</sub>(SO<sub>4</sub>)<sub>3</sub> can be made by heating pure FeSO<sub>4</sub>.7H<sub>2</sub>O with a large excess of H<sub>2</sub>SO<sub>4</sub>. The hot H<sub>2</sub>SO<sub>4</sub> dehydrates and oxidizes the FeSO<sub>4</sub>, yielding cryst. Fe<sub>3</sub>(SO<sub>4</sub>)<sub>3</sub> which can be filtered on asbestos, washed with alc. and then with ether and dried to const. wt. in a water oven. This Fe<sub>3</sub>(SO<sub>4</sub>)<sub>3</sub> serves as a good standard for KMnO<sub>4</sub>, being reduced with Zn in H<sub>2</sub>SO<sub>4</sub> soln. and titrated in the usual way.

PERCY H. WALKER.

New Method of Preparation of Nitrous Oxide and Application to the Analysis of Nitrates. A. QUARTAROLI. Lab. chim. r. ist. tech. Viterbo. Gazz. chim. ital., 41, II, 53-9; see C. A., 5, 3210. Chas. A. ROUILLER.

A Source of Error in the Gasometric Determination of Nitrates and Nitrites According to Schloesing and Piccini Respectively. O. RUFF AND E. GERSTEN. Z. anorg. Chem., 71, 419-26.—The effect of the presence of arsenites and sulfides was investigated. As<sub>2</sub>O<sub>3</sub> in HCl soln. always causes loss of NO giving low results for nitrates. The greater the amt. of As<sub>2</sub>O<sub>3</sub> and the smaller the HCl conc. the greater the loss. The reduction in neut. or alk. soln. is not so great as in acid soln. With nitrites the reduction and consequent loss is immaterial in neut., but large in acid soln. The error caused by As<sub>2</sub>O<sub>3</sub> in nitrate detn. is partially due to reduction to nitrite. The presence of H<sub>2</sub>S gives low results with nitrates and nitrites in acid soln. Several tables are given showing amts. of NO and N evolved and the effects of diff. concs.

E. W. BOUGHTON.

The Determination of Nitric Nitrogen in Gun Cotton, in Nitroglycerin and other Analogous Products. H. Pellet. Ann. chim. anal., 16, 294-6.—The method of Schloesing contains several sources of error: (1) CO<sub>2</sub> generated by the action of HCl on chalk is never pure; (2) air is liberated by the action of the HCl; (3) NO reacts with caustic KOH to give different products. These errors partially compensate each other. P. places the gun cotton in a flask with some dist. H<sub>2</sub>O and expels the air by b. The soln. of ammoniacal FeSO<sub>4</sub> and HCl is then run in and the reaction proceeds regularly. Boiled dist. H<sub>2</sub>O is used in the bath holding the graduated tubes for receiving the gas. The use of CO<sub>2</sub> and KOH is thus eliminated. The app. is tried out with a titrated soln. of NaNO<sub>3</sub> or KNO<sub>2</sub>, reducing the amt. of gas obtained to 0° and 760 mm. For substances like nitroglycerin, the procedure is changed. The FeSO<sub>4</sub> soln. and HCl are placed in the flask and the air expelled by b. The material to be analyzed is then run in slowly. The Schloesing method, as thus modified, is the simplest and most accurate for this work.

Examination of Material Containing Copper, Nickel and Cobalt. H. PEDERSEN. Metallurgie, 8, 335.—The author takes 2-5 g. of pyrrhotite, 0.5-2.5 g. of Ni mat and 10-20 g. of slags. After dissolving in conc. HNO<sub>3</sub>, conc. H<sub>2</sub>SO<sub>4</sub> is added and the soln. evapd. till fumes of SO<sub>2</sub> are evolved; H<sub>2</sub>O is added and the evapn. repeated. The residue is filtered off and the Fe pptd. 3 times with NH<sub>4</sub>OH after which it is titrated. H<sub>2</sub>SO<sub>4</sub> is added to the filtrate from the Fe(OH)<sub>3</sub>, and after reducing the vol. to 150 cc. the Cu is detd. electrolytically. Then the soln. is neutralized with NH<sub>4</sub>OH and 20 cc. of the conc. reagent added in excess and then the Co and Ni are detd. electrolytically. The Ni and Co are dissolved by b. with 40 cc. conc. HNO<sub>3</sub>, the soln. evapd. to dryness, 1 cc. conc. HNO<sub>3</sub> added and the soln. again evapd. on the H<sub>2</sub>O bath to remove all HNO<sub>3</sub>. This residue is treated with a few drops of dil. HCl, dissolved in 150 cc. H<sub>2</sub>O and the Ni pptd. with 20 cc. of 1% dimethylglyoxime in alc. The ppt. is filtered on a Gooch crucible, washed 6 times with hot H<sub>2</sub>O, dried 2 hrs. and weighed; it contains 20.3% Ni.

WILLIAM T. HALL.

The Alkalimetry of Magnesium Ammonium Phosphate and the Acidimetry of Ammonium Phosphomolybdate. A Proposed Simplification of the Analysis of Products Containing Phosphates and Phosphorus. I. FRANZ HUNDESHAGEN. Stuttgart, Z. offent. Chem., 17, 283-94.—The procedure is as follows: The pptd. MgNH<sub>4</sub>PO. which should contain from 0.16-0.20 g. P<sub>2</sub>O<sub>4</sub> is filtered on a Gooch and washed first with the smallest possible amt. of 21/2% NH4OH and then with 95% neutral alc. until NH, is removed. A drop of alc. rosolic acid (1: 200) may be added to the ppt. to test for NH<sub>2</sub>. If this is not colored red, but remains clear yellow no NH<sub>4</sub> remains, and the indicator may be removed by a little more alc. The ppt. is now completely returned to the pptg. vessel which must also be free from NH<sub>2</sub>. <sup>1</sup>/<sub>2</sub>-1 cc. of methyl orange soln. (1-10,000) is added and 0.1 N acid (HCl, H<sub>2</sub>SO<sub>4</sub> or HNO<sub>2</sub>) is run into the beaker containing the suspended ppt. until 2-5 cc. excess of acid have been added. The excess of acid is titrated with 0.1 N Na<sub>2</sub>CO<sub>2</sub>. 1 cc. 0.1 N acid = 0.001552 g. P or 0.003552 g.  $P_0O_0$  according to the equations MgNH<sub>2</sub>PO<sub>4</sub> + 2HCl = NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub> +  $MgCl_2$  or  $2MgNH_4PO_4 + 4HCl = Mg(H_2PO_4)_2 + MgCl_2 + 2NH_4Cl$ . This method is easy of operation, much more rapid than the grav. and gives results which agree very closely with the grav. as shown by the author's results. The presence of SiO<sub>2</sub> does not interfere with the detn. as in the grav. method. The error caused by the presence of As is less than in the latter method. When desirable, As may be readily separated from the mixt. of arsenate and phosphate, and the latter repptd. and detd. A method similar to the foregoing may be applied to the detn. of As. 1 cc. 0.1 N acid = 0.00375 g. As or 0.00575 g. As  $Q_1$ . In the detn. of As, the ppt. is washed with a mixture of  $2^1/2$ NH<sub>4</sub>OH and alc. (1:1) instead of NH<sub>4</sub>OH alone. P. B. DUNBAR.

Alkalimetry of Magnesium Ammonium Phosphate and Acidimetry of Ammonium Phosphomolybdate. II. F. Hundeshagen. Z. öffent. Chem., 17, 302-9; cf. preceding abstr.—According to the equation  $(NH_4)_2(MoO_2)_{12}PO_4 + 23NaOH = (NH_4)_2MoO_4 + 11Na_2MoO_4 + NaNH_4HPO_4 + 11H_2O$  each cc. of N alkali corresponds to 0.0013496 g. P or 0.0030887 g. P<sub>2</sub>O<sub>4</sub>. The actual factors differ slightly, especially when phenolph. is used because of the weakly basic NH<sub>2</sub>. In the method 1 cc. N alkali equals 0.001345 P or 0.003078 P<sub>2</sub>O<sub>4</sub>. The yellow phosphomolybdate is pptd. at 80-90° in a soln. containing about 5% NH<sub>4</sub>NO<sub>2</sub> and 26-80 mols. free HNO<sub>2</sub> to one of PO<sub>4</sub>. Details for satisfactory pptn. are given. The ppt. is redissolved and thrown down as usual. After filtering and washing with 2% KNO<sub>2</sub> soln. it is suspended in H<sub>2</sub>O and titrated with standard alkali using phenolph. as indicator. The advantages over the grav. method are rapidity and the use of small amts. of material.

D. S. PRATT.

Titration of Phosphoric Acid. M. WAGENAAR. Utrecht. Pharm. Weekblad, 48, 845–50.—W. points out the 3-fold character of the dissociation of H<sub>2</sub>PO<sub>4</sub>: (a) H<sup>+</sup>, H<sub>2</sub>PO<sub>4</sub><sup>-</sup>,

(b) H<sup>+</sup>, HPO<sub>4</sub><sup>-</sup>, (c) H<sup>+</sup>, PO<sub>4</sub><sup>-</sup> which is very weak. (a) may be titrated with Me orange, (b) with phenolph. (c) may be made apparent by adding Pb(NO<sub>2</sub>)<sub>2</sub>, e. g., 3Pb(NO<sub>2</sub>)<sub>2</sub> + 2Na<sub>2</sub>HPO<sub>4</sub>  $\Longrightarrow$  4NaNO<sub>2</sub> + Pb<sub>2</sub>(PO<sub>3</sub>)<sub>2</sub> + 2HNO<sub>2</sub> but Pb<sub>2</sub>(PO<sub>4</sub>)<sub>2</sub> is sol. in HNO<sub>3</sub>, and consequently the reaction is not final; if NaOH is added it will be. Hence after titrating with Me orange and phenolph. add Pb<sub>2</sub>(PO<sub>4</sub>)<sub>2</sub> and titrate the rose-red soin. to yellow with NaOH.

V. E. Henderson.

Apparatus for Determining Carbon (BUTZBACH, FENNER). 1.

Determination of Copper in Preserves (SERGER). 12.

Determination of Sugar in the Blood (FORSCHBACH, SEVERIN). 11.

Sugar in Urin (DE JAGER). 11.

Determination of Nitrates in Molasses (PELLET, MÜLLER). 28.

Treatment of Anode Sludge (KERN). 4.

Estimation of Iron with Iodine (ROMIJN). 17.

Detection of Nitrates and Nitrites by Hydrostrychnine (DENIGES). 14.

Apparatus for Specific Gravity of Gases (GULICH). 1.

New Gas Analysis Apparatus. 1.

Gas Analysis by Fractional Combustion (UBBELOHDE, CASTRO). 21.

### 8. MINERALOGICAL AND GEOLOGICAL CHEMISTRY.

#### ROGER C. WELLS.

Fayalite from the Island of Pantelleria. J. SOELLNER. Freiberg. Z. Kryst. Min., 40, 138-51.—Fayalite crystals occur as a subordinate constituent of the sands in and about the crater Cuddea Mida on Pantelleria. The chief constituents of the sand are cossyrite, quartz and feldspar. In the augite andesite fayalite occurs only as the earliest generation of intratelluric crystn. and often shows later resorption. The common habit is short prismatic along the c axis with hexagonal appearance. A second type is similar to the first but the crystals are somewhat tabular, parallel to (010). The third type is tabular after (010) and elongated along the a axis. The forms are b(010), m(110), s(120), k(021), c(001), e(111), d(101). The crystals are of orthorhombic symmetry. a:b:c:=0.46000:1:0.58112. Cleavage 010 good, (001) moderately good. H 6.5-7. The crystals are transparent to translucent. Color, wine-yellow to greenish yellow. Luster, vitreous. The indices of refraction (as measured by several oriented prisms) are given. The optic orientation is: a = 2; b = X; c = Y. The optic character is negative  $\rho > V$ . The pleochroism of thick plates is considerable and for a plate 0.25 mm. thick X = orange-yellow, X = Z = greenish yellow. In sections 0.03 mm. thick, the absorption is perceptible and Y is perceptibly yellowish while X and Z are bluish yellow with a touch of green. This distinguishes it from olivine which is colorless. The mineral is completely sol. in cold conc. HCl with the formation of gelatinous SiO<sub>2</sub>. It easily fuses to a black magnetic glass. The following analysis was made by Dittrick: SiO, 28.89; FeO, 1.19; FeO, 5.08; FeO 56.05; MnO 3.39; CaO 0.74; MgO 3.11; alkalies 0.42; H<sub>2</sub>O 1.07; total 99.94; sp. gr. 4.24. E. S. LARSEN.

Some Calcite Twins. V. GOLDSCHMIDT AND R. SCHRÖDER. Heidelberg. Z. Kryst. Min., 49, 133-4.—Calcite crystals from Fiesch, Rhone Thal, and from Reckingen, Rhone, show cross twinning after  $\delta = -1/2$ . A crystal of calcite from Egremont shows juxtaposition twinning after  $\varphi = -2$ . E. S. LARSEN.

Calamine, Catapleiite and Diopside. V. Goldschmidt and R. Schröder. Heidelberg. Z. Kryst. Min., 49, 135-7.—The calamine from Altenberg, near Aachen, described by W. Schulze (Mitt. Nat. Ver. New-Vorpomm. u. Rug., 18, 59 (1887)) as having the new form (205) does not appear to have that form. Catapleiite does not appear to have the form (1.0.I.30) described by J. Gotz (Mitt. Nat. Ver. Vorpomm. u. Rug., 18, 57 (1887)). The diopside from Alabama, described by J. Gotz (Mitt. Nat. Ver. Vorpomm. u. Rug., 18, 54 (1887)) is twinned after (100) and the new forms (15.4.0), (15.0.4) and (15.4.4) described by Gotz do not occur while (411) and (401) are present. Other corrections are made.

Corundum. V. GOLDSCHMIDT AND R. SCHRÖDER. Heidelberg. Min. petrog. Mitt., 29, 461-88.—The authors describe 11 crystals of sapphire from Ceylon and 4 crystals of ruby from Burma.

E. S. LARSEN.

The Identity of Stelznerite and Antierite. W. T. Schaller. Washington. Z. Kryst. Min., 49, 9-10.—Antierite from the original locality described by Hillebrand occurs in minute crystals with a short prismatic habit. The crystals are bluish green in color and the thicker individuals are perceptibly pleochroic, showing blue-green in the long direction and yellowish green normal thereto. The extinction is parallel; the birefringence is high. All of these properties agree with those of stelznerite. The sp. gr. of antierite is 3.93 and that of stelznerite, with some admixt. of gypsum, is 3.884. The formula given by Hillebrand for antierite is 10CuO<sub>4</sub>,SO<sub>4</sub>,7H<sub>2</sub>O while that for stelznerite is 3CuO,SO<sub>4</sub>,2H<sub>2</sub>O. The difference is slight and the 2 minerals are no doubt identical with the formula 3CuO,SO<sub>4</sub>,2H<sub>2</sub>O. The name antierite has the priority over stelznerite.

A Hitherto Unnoticed Chemico-crystallographic Relation. F. LOEWINSON-LESSING. St. Petersburg. Centr. Min., 1911, 440-2.—While it is well known that a double compd. may have lower symmetry than its constituents (cf. calcite and dolomite) and this holds for hydrous compds., the union of a silicate and a non-silicate is here pointed out as favoring higher symmetry. This is exhibited in the sodalite group and in marialite, helvite, danalite, etc.

E. T. Wherry.

Schaumopal. Otto Hauser. Tech. Inst., Univ. Berlin. Centr. Min., 1911, 436-8.—Near the Virunga volcano, German East Africa, much SO<sub>2</sub> is exhaled, which, uniting with H<sub>2</sub>O and O<sub>2</sub>, condenses as H<sub>2</sub>SO<sub>4</sub> on the lava. This has at several points been decomposed into a porous white mass with the comp. of a very impure opal, containing up to 8.20% H<sub>2</sub>O, for which the name schaumopal is proposed. E. T. W.

Mineralogy of the Magnesite Deposits of Eichberg, Semmering; Eichbergite, a New Sulfantimonate. O. Grosspietsch. Leoben. Centr. Min., 1911, 433-5.—In addition to the previously known minerals associated with the magnesite—stibnite, tetrahedrite, tennantite, and pyrite—there have been now found galenite, rumpfite, comp. SiO<sub>2</sub> 30.71, Al<sub>2</sub>O<sub>3</sub> 38.10, Fe<sub>2</sub>O<sub>3</sub> 1.83, MgO 17.83, H<sub>2</sub>O 11.30, sum 99.77; and a gray-green glassy mineral, weathering dull gray, with H = 6 + and sp. gr. 5.36 for which the name Eichbergite is proposed. Its comp. is Cu 3.62, Fe 1.45, Bi 51.53, Sb 30.00, S 12.74, sum 99.34, giving (Cu,Fe)Bi<sub>2</sub>Sb<sub>2</sub>S<sub>3</sub>.

E. T. Wherry.

Remarks on O. Mügge's Paper on Ferruginous Quartz from Suttrop. R. Brauns. Min. Inst., Univ. Bonn. Centr. Min., 1911, 465-6.—M.'s views on the formation of the ferruginous quartz crystals described in C. A., 5, 2384, really agree with those of the author. Chalcedony is undoubtedly identical with quartz, being fibrous because of growth from solid gels. The crystals in question are well explained by the lowered stability of the quartz containing iron oxide, which permitted its replacement by chalcedony, from which in turn pure quartz formed.

E. T. Wherry.

Variation in the Optical Axial Angle of Gypsum with Increasing Temperature.

R. Brauns. Min. Inst., Univ. Bonn. Centr. Min., 1911, 401-5.—The supposedly extremely accurate measurements of Tutton (C. A., 5, 1512) of the temp. at which gypsum becomes uniaxial (104-113°) must be affected by a constant error, since B. can demonstrate the phenomenon by heating on a water bath. T's specimens were probably not warmed to the center when the temp. was read.

E. T. Wherry.

Chemico-mineralogical Studies on Glaucodot. A. BEUTELL. Min. Inst., Univ. Breslau. Centr. Min., 1911, 411-5.—Following up his expts. on arsenopyrite (C. A., 5, 2795) the author tried glaucodot with the comp. S, 20.35, As 38.80, Fe 21.83, Co 16.36, Ni 0.46, Cu 1.93, sum 99.73. On heating in the vacuum only a small part of the As could be distilled out; it is therefore united to the Co, giving the formula:

Oncosine from Variney, Aosta. Guiseppe Pioliti. Atti accad. sci., torino, 44, 1-4; Neues Jahrb. Min. Geol., 1910, II (ref.) 359.—Green masses in calcite had the sp. gr. 2.819 and comp.: SiO<sub>2</sub> 50.98, Al<sub>2</sub>O<sub>3</sub> 25.63, FeO 0.35, MgO 6.31, K<sub>2</sub>O 12.32, Li<sub>2</sub>O tr., H<sub>2</sub>O 4.14, sum 99.73. It differs from cossaite in paler color and greater fusibility and is most closely related to the oncosine of Kobell.

E. T. Wherey.

Sodium Sanidine from Mitrowitza. Correction. Franz Angr. Graz. Centr. Min., 1911, 424.—The orientation of the mineral described in C. A., 5, 1383, was erroneous, and it is in no sense a new species.

E. T. W.

The Composition of Jamesonite and Warrenite. W. T. SCHALLER. Z. Kryst., 48, 562-5.—From a review of the published analyses, it is concluded that the formula of jamesonite is 4PaS.FeS.3Sb<sub>2</sub>S<sub>2</sub> as first proposed by Loczka. Warrenite is a mixt. of jamesonite with zinckenite.

W. T. SCHALLER.

Dimorphous Natrolite. St. J. Thugutt. Warsaw. Centr. Min., 1911, 405-11.

—Analysis of a natrolite occurring in typical monclinic crystals in a phonolite from Schömitz, near Karlsbad, gave SiO<sub>2</sub> 41.34, Al<sub>2</sub>O<sub>3</sub> 23.49, CaO 0.04, Na<sub>2</sub>O 14.71, H<sub>2</sub>O 9.03, comptonite, intimately mixed, 12.15, sum 100.76. This gives the normal ratio, yet yields abnormal effects on standing with methylene blue, etc., so must be a metameric variety, and for it the name epinatrolite is proposed. It is derived from minerals of the sodalite group, and is more readily decomp. by heat than ordinary natrolite, which is formed from nephelite. Its monoclinic symmetry, with extinction angle 5-6°, can not be due to morphotropic intergrowth of a K mineral, for only traces of K are present. This is probably the true system of natrolite, the apparent rhombic crystn. of some specimens being explainable by repeated twinning. E. T. Wherry.

Agate Problems. RAPHAEL E. LIESEGANG. Edinger's Neurolog. Inst., Frankfurt. Centr. Min., 1911, 497-507.—It seems certain that most agates are formed by diffusion of Fe oxide into SiO<sub>2</sub> gels (cf. C. A., 5, 1384). The original gels are not solid opal, however, but a more hydrous, soft, substance. The Fe is either originally distributed uniformly through the gel, or enters as a colloidal soln. from the surrounding rocks, and its pptn. is probably caused by Na or K silicates adsorbed by the SiO<sub>2</sub>. As water is lost the gel contracts, at first from the outside, later, when a solid layer has formed there, from the inside, a cavity being produced in which quartz crystals may form, or a new mass of gel be introduced. This dehydration of the gel is due to decrease of adsorption surface attendant on the uniting of SiO<sub>2</sub> particles in obedience to the principle of the growth of large individuals at the expense of small ones. The single chalcedony fibers often observed to traverse a number of colored bands are clearly the result of such secondary growth. The usual assumption that colloids like opal are amorphous is hardly justified under our present conception of colloid phenom-

ena; they are simply ultra-microcrystallin, and the fibers which grow are the more favorably situated ones, according to the above principle. The horizontally banded agates represent, however, a different situation, being really stratified, and produced by intermittent introduction of small portions of gel into a cavity. E. T. Wherey.

New Automatic Mercury Air Pump, and Distillation of Arsenopyrite, Glaucodot and Cobaltite in the Cathode-ray Vacuum. A. BEUTELL. Min. Inst., Univ. Breslau. Centr. Min., 1911, 491-5.—Description, with fig., of the pump (cf. C. A., 5, 1210) and the technic of the method of studying the minerals (cf. C. A., 5, 2795).

E. T. Wherry.

Geology and Mineral Resources of the Nizina District, Alaska. F. H. MOFFIT AND S. R. CAPPS. U. S. Geol. Survey, Bull. 448, 58 pp.—Describes the region containing the Kennicott Bonanza Cu mine and the placers of Dan and Chititu creeks. Four important geologic formations are represented on the map. They are, beginning with the oldest, the Nikolai greenstone, the Chitistone limestone, the McCarthy shale, and the Kennicott formation. The last 2 are of Upper Triassic age and are sepd. by an unconformity from the Kennicott formation which is of Upper Jurassic age, possibly extending into the Lower Cretaceous. A large part of the shales which were considered by earlier workers as belonging to the McCarthy shale is in reality part of the Kennicott formation. The Nikolai greenstone, a succession of lava flows, is the source of the Cu deposits, the most valuable Cu deposits in this region being situated near or at the contact between the Nikolai greenstone and Chitistone limestone. Some Cu even extends into the limestone to a distance of 1,000 feet. An important fact concerning the Cu ores is that they are connected with faults and shear sones, many of which, though difficult to recognize, give opportunity for the circulation of Cu-bearing waters. The Bonanza mine is a large deposit of chalcocite located in such a shear zone. The placer deposits are shown to be derived from small quartz veins in the Kennicott shales and are considered to be connected in origin with the numerous quartz porphyry dikes that cut the shales. R. C. WELLS.

Differentiation in the Southern Black Forest. PAUL NIGGLI. Petr. Inst., Polytechn. School, Zurich. Centr. Min., 1911, 438-40.—A granit magma presents extensive differentiation phenomena.

E. T. Wherey.

Permo-triassic Sandstones and Eruptive Sheets from Northern Uruguay. KARL. WALTHER. Secc. Min. Geol., Inst. Agronom., Montevideo. News Jakrb. Min. Geol., (Beil. Bd.), 31, 575-609.—In addition to acid and basic eruptives a spherulitic vitrophyre altered into chalcedony and quartz has been observed.

E. T. W.

The Eruptive Rocks of the Habichtswald, Kassel, and its Foothills. KARL SCHLOSS-MACHER. Frankfurt. News Jahrb. Min. Geol., (Beil. Bd.), 31, 641-83.—Includes a new analysis of trachydolerite.

R. T. W.

Petrographic Description of the Basalts of Langenberg and Vicinity, North of Fritzlar. RUDOLF BERNGES. Hanau. News Jahrb. Min. Geol., (Beil. Bd.), 3x, 610-40.—Analyses.

E. T. W.

The Iron Deposits of Bilbao. John. Halle. Z. prakt. Geol., 19, 208-12.—
Hematite and siderite ores occur as a replacement of limestone, resting on massive sandstone which acted as an impervious basement.

E. T. Wherey.

The Coal Fields of the Gondwana Formation, India. F. Schreiber. Z. prakt. Geol., 19, 169-203.—Analyses. E. T. W.

Iron Ores. Occurrence and Composition. J. Iron Steel Inst., 83, 514-25.—Abstracts of numerous papers from all over the world.

R. C. W.

The Mount McKinley Region, Alaska. A. H. BROOKS, Descriptions of the

Igneous Rocks. L. M. PRINDLE. U. S. Geol. Survey, Prof. Paper 70, 234 pp., maps.— This paper describes the exploration in 1902 of a vast unknown tract north of Mt. McKinley. It also deals with the geologic relations and mineral resources of the region. The igneous rocks are in their character, associations, mode of occurrence, and age similar to rocks in the western part of the U. S. and Canada. They present an illustration of the uniformity of types produced by differentiation in widely sepd. regions and also of the wide variation possible in individual types within small areas. Most of them are comparatively fresh, but protoclastic phenomena are exhibited by some of the olivine-pyroxene monzonites, where the pyroxenes have been granulated to a greater or less extent before the final consolidation of the rock, and cataclastic phenomena are shown in the minute faulting and partial granulation of the quartz in quartz diorites collected near Mt. McKinley. The results of an advanced stage in the metamorphism of an igneous rock are shown in the rhyolitic schists of the northern foothills of the Alaska Range. The wide distribution of the igneous rocks, the great altitudes attained by many of them, and their occurrence at several geologic horizons all bear witness to a great amt. of igneous material, to long-continued igneous activity, and to the great structural influence of these rocks. The mineral resources are chiefly Au, Cu and coal deposits. R. C. WELLS.

Pliocene Lignite in the Bacau District, Roumania. C. M. Dozy. Min. Soc., Bacau. Z. prakt. Geol., 19, 203-8.—Analysis. E. T. W.

Peculiarities of the Gold Deposits of the Schlangenberg Type, Altai, Siberia. GREGOR MAYER. St. Petersburg. Z. prakt. Geol., 19, 234-9.—The ores occur in fissures in schist and hornstone, and have probably been derived from the underlying porphyry.

E. T. Wherry.

Deposits of Crystallized Gold in Limestone at Totok, Celebes. H. AND A. HIRSCHI. Z. prakt. Geol., 19, 213-4.—Waters given off from eruptive rocks have circulated through fissures in limestone, depositing on their walls a layer of quartz crystals, then quartz calcite and plagioclase feldspar nearly simultaneously, and finally Au, 680 fine, the remainder being Ag, in dendritic-crystallin crusts. E. T. Wherey.

Separating Apparatus (DREIBRODT). 1.

Diffusion of Petroleum through Fuller's Earth (GILPIN, BRANSKY). 22.

Coloring of Inorganic Substances by Radium (NIEDERSTADT). 3.

Age of the Earth (JOLY). 3.

# 9. METALLURGY.

# WILLIAM BRADY, WILLIAM T. HALL.

The Smelting of Copper Mat Rich in Lead. ROBERT HESSE. Metallurgie, 8, 321-35, 365-75.—The expts. show that it is impossible to effect the removal of Pb by a simple-reaction smelting of the roasted and unroasted mat. If the O conc. is large enough to cause the sepn. of methyl, a Cu-Pb alloy is always obtained. On the other hand, if the roasted mat, or a mixt. of the roasted and unroasted mat, is melted with the addition of some SiO<sub>2</sub>, a nearly pure Cu is obtained and the Pb and Fe form silicates. In this case too low O conc. results in the formation of a conc. Cu mat, low in Pb, together with black Cu. The process is an economical one provided the Pb and Cu content of the original mat is not too low. William T. Hall.

Chemical Study of the Cyanide Leaching of Silver Ores. E. KÜHN. *Metallurgie*, 8, 399–404, 421–36, 464–72, 481–92.—The chem. reactions involved in the Patera, Kiss,

Russel, Russel-Patera, and Cyanide-Patera processes are outlined, together with the advantages and disadvantages of each. The reactions are studied carefully from a physico-chem. standpoint and expts. with ore made. To obtain a leaching liquor with max. solvent power, the conc. of the sulfide S must be kept as low as possible. This is effected by oxidation with air and various oxidizing agents whereby Na<sub>2</sub>S is changed to Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> and finally to Na<sub>2</sub>SO<sub>4</sub>, blowing with an indifferent gas to get rid of H<sub>2</sub>S, and by pptn. with Pb and Zn salts. Lime must be added to keep the soln. alk., to make possible the blowing, and to permit the oxidation in spite of the introduction of OH ions. In treating a Mexican ore bearing 1.8% Ag as Ag<sub>2</sub>S, with gang chiefly SiO<sub>2</sub> and CaCO<sub>3</sub>, treatment according to the author's ideas gave better extraction in shorter time than could be obtained with the aid of the Just process. In the development of new extraction app. the aim should be to attain mechanical movement and only as much air as is absolutely necessary; the Pachuca tank seems to fulfil very nearly this last requirement.

WILLIAM T. HALL.

Damascus Steel. N. T. Belair. Metallurgie, 8, 449-56, 493-7.—An outline of the work that has been done in studying the nature of, and toward duplicating Damascus steel.

WILLIAM T. HALL.

Slime Thickening at El Tigre. D. L. H. FORBES. Eng. Mining J., 92, 691-2.—Two continuous thickeners of the Dorr type, but with specially designed spiral paddles, are used at El Tigre mill.

ROBERT KANN.

Concrete Jigs at Cananea. F. J. STRACHAN. Eng. Mining J., 92, 698-9.

R. K.

De Wilde Precipitation Process. G. WITTEVEEN. Mines and Minerals, 31, 342-3.—The process is executed in 3 stages. (1) The alkaline soln. containing Au and Ag as cyanides is acidified with dil. H<sub>2</sub>SO<sub>4</sub> containing NaCl. The AgCl pptd. is sepd. by a filter press. (2) To the filtrate Cu<sub>2</sub>Cl<sub>2</sub>, dissolved in NaCl soln., is added. The Au in soln. is co-pptd. with the Cu<sub>2</sub>(CN)<sub>2</sub>. About 7 times as much Cu<sub>2</sub>(CN)<sub>2</sub> as of Au is necessary for the complete pptn. of the latter. After agitation the ppt. is filtered off and the Au recovered. (3) Most of the CN is regenerated by rendering the final soln. alkaline. Stages 2 and 3 may be combined. Lab. expts. prove that the loss of CN by volatilization from acid soln. is much greater with increased conc. The loss of KCN for a 0.05% soln. is 50 g. per ton and 375 g. for a 0.4% soln.

ROBERT KANN.

Cyanidation of Cripple Creek Ores. PH. ARGALL. Mining Sci. Press, 101, 804-5.

—Cyanide practice and results in this district are reviewed and further prospects considered, particularly with reference to milling low grade sulfotelluride ores.

ROBERT KANN.

Cyanidation of Concentrate. G. C. Evans. Mining Sci. Press, 101, 844-5.

—At Kuk San Dong, Korea, Au-bearing sulfide ores, containing much arsenopyrite are crushed to 15-mesh size. 6-9 lbs. lime per ton are mixed with the concentrate obtained. The usual trouble of lime coating on the Zn in the pptn. boxes is obviated by preliminary settling in lumps. The concentrate assays \$9-10 per ton and 65% cyanide extraction is obtained.

ROBERT KANN.

Improvements in Silver, Lead and Copper Smelting. L. S. Austin. *Mining Sci. Press*, 102, 63-6, 176-8.—Advances made during 1911 are considered. The advantages and disadvantages of basic as compared to acid converters are enumerated.

ROBERT KANN.

Empire Mines Cyanide Plant. F. C. LEADGUTH. Mining Sci. Press, 102, 237.
R. K.

Recent Cyanide Practice at the Montezuma Abbé Engineering Co. Mining Sci.

Press, 102, 271.—A reply is made to a statement that in the use of a spiral feed in tube mill practice, clogging resulted. It is affirmed that this condition is due to the fact that the pulp carried only 37% moisture. In most plants the soln. used is 1-1 and the pulp should never contain less than 40% moisture. ROBERT KANN.

Air Lift Agitation of Pulp Slime. C. TOOMBS. J. Chem. Met. Soc. S. Africa, 11, 614.—When slime is treated with sufficient lime to neutralize the FeSO<sub>4</sub> and H<sub>2</sub>SO<sub>4</sub> the former is pptd. as Fe(OH)<sub>2</sub> which at once absorbs all the O in the slime, and only a small % of which is changed into Fe(OH)<sub>2</sub>. Air lift agitation quickly introduces more O and at once changes the Fe(OH)<sub>2</sub> into Fe(OH)<sub>3</sub> and introduces the excess of O which is necessary for perfect conditions in cyaniding.

E. C. L.

Gold Milling in California. W. H. Storms Met. Chem. Eng., 9, 493-6.

J. J. M.

Etching of Names, Figures, etc., on Steel. E. Elektrochem. Z., 18, 145.—The surface is first covered with a carefully prepared mixt. of 2 parts powdered asphalt, 1 part rosin, 1 1/2 part wax and a little tallow. A very thin coating of this is put on the surface to be etched and after fixing the design, the etching is done with HNO<sub>2</sub>, acetic or tartaric acid.

W. E. RUDER.

The Schoop Process for Metal Coating. PRADEL. Elektrochem. Z., 18, 23-4.

—P. reviews the older processes for powdering and spraying metals and other substances and shows how they differ from the present method.

W. E. R.

Actual State of Our Knowledge Regarding the Process of Cementation of Steel. F. GIOLITTI. Rass. min., 35, 100-6, 125-30, 141-5. Chas. A. ROUILLER.

The Influence of Galvanizing on the Strength of Wire. H. WINTER. Iron Age, 86, 502; see C. A., 5, 64.

L. A. TOUZALIN.

The Iron and Steel Industry of Mexico. John Birkinbing. Iron Age, 86, 556-68.

L. A. T.

The Iron Ore Resources of the World. L. DE LAUNAY. Iron Age, 86, 674-6.—The author considers the 3 factors: (1) Metallurgical treatment, (2) changes in methods of transportation, (3) shifting of com. centers and the creation of new ones. L. A. T.

The Introduction of the Bessemer Process in America. Geo. W. MAYNARD. Iron Age, 86, 746.—Historical. L. A. T.

Preventable Blast Furnace Accidents. R. H. Sweetser. Iron Age, 86, 1022-3.
L. A. T.

The British Steel Maker's Ore Supplies. W. H. HERDSMAN. Glasgow. Iron Age, 86, 1270-4.—A review.

L. A. T.

Iron Ore and Flue Dust Briquetting. Anon. Iron Age, 86, 1330-1.—A brief review and a short illustrated description of the Ronay process.

L. A. T.

The Burgers Thin Wall Blast Furnace. Anon. Iron Age, 86, 1431.—The Burgers furnace is described. Satisfaction with the construction and regular working of the furnace is expressed; the coke consumption is low.

L. A. T.

Continuous Cyanidation. JOHN V. N. DORR. Met. Chem. Eng., 9, 436.—The important point in continuous cyanidation is the passing of the slime and its successive use for the diln. of the increasingly rich pulp as the soln. passes toward the head of the series. Pptn. is then necessary only after it has been several times enriched, thus reducing the amt. to be pptd. to a small fraction of that otherwise required. Mr.

Rothwell's flow sheet (cf. C. A., 5, 2798) should be of great value to the cyaniding fraternity. Figures are given to show the % of recovery of dissolved values with the pptn. of a relatively small amt. of soln. which would be obtained should the method described be used.

L. A. T.

The Action of Salt Solutions and of Sea Water on Iron at Various Temperatures. J. A. N. FRIEND AND J. H. BROWN. J. Chem. Soc., 99, 1302-6; Proc. Chem. Soc., 27, 156.—By exposing thin plates of pure Fe to NaCl solns., it was found that even the dil. solns. cause less corrosion than pure H<sub>2</sub>O if the temp. is above 13° but below 13° the dil. NaCl solns. cause more corrosion. At 13° the corrosive effect is practically the same for ordinary sea H<sub>2</sub>O as for dist. H<sub>2</sub>O.

W. T. H.

Ferrotitanium. G. B. WATERHOUSE. *Met. Chem. Eng.*, 9, 437.—Fe-Ti alloys containing 6-7.5% Ti do not contain more than 0.25% combined C. Apparently Ti decomposes Fe<sub>2</sub>C setting free graphite. W. T. H.

A New Method for Determining the Degree of Rusting. H. JACOB AND R. KARS-BOHRER. Chem. Ztg., 35, 877-8.—The tared piece of rusted Fe is suspended as cathode in a suitable electrolyte; as anode, a Pt or C electrode may serve. On passing the current through the cell, the rust is dislodged by the H evolved. A suitable electrolyte is 0.25% Na<sub>2</sub>SO<sub>4</sub> soln. The author uses a current of 0.5 to 4 amperes and dets. the degree of corrosion by weighing the metal after the expt.

W. T. H.

The Crystallization of Cast Metals. C. H. DESCH. Metal Ind., 9, 376-7.—The author describes the crystallizing of molten metals and explains how the crystn. can be studied microscopically.

W. T. H.

Copper-Nickel Alloys. A. H. HIORNS. Metal Ind., 9, 387-90.—These alloys form a series of solid solns. with no compds. or eutectics. After annealing, the elastic limit of the alloys with less than 31% Ni is very low. When 15% Ni is exceeded, the tenacity is increased but the elongation and reduction of area are reduced and the hardness increased. Alloys with more than 50% Ni are magnetic but the magnetic property ceases at a certain critical temp., this temp. being lower the greater the % Cu. The magnetic solid soln. is termed "alpha" and the non-magnetic soln. "beta."

W. T. H.

Binary Metallic Alloys. XIII. K. BORNEMANN. Metallurgie, 8, 270-80, 289-95.—A summary of the literature on the alloys of Sn with Na, K, Cu, Ag, Au, Mg, Ca, Zn, Cd, Al, Tl, Pb, Bi, Cr, Mn, Fe, Co, Ni and Pt. WILLIAM T. HALL.

The Influence of the Condition of the Surface on the Rusting of Iron. K. Arnot. Metallurgie, 8, 353-8.—The author explains the fact that when rusting begins it spreads with increasing rapidity, by the galvanic action between the rust and the metal. If the surface is rough or porous the rust adheres easily, thus forming more couples and causing rapid corrosion. This explains why some surfaces are quickly covered with rust while others rust only in spots. A surface covered with rust is not protected from further corrosion because the coating is porous and the O of the air easily penetrates it. Dampness hastens corrosion because the rust absorbs moisture like a sponge and thus allows the O to diffuse through it readily. For this reason corrosion is very rapid when Fe is placed in sand which easily transmits O and at the same time keeps the surface moist. Corrosion is hastened if the soil contains organic matter. The rapidity of corrosion of steel depends on the scale left by hammering or rolling. If the scale adheres, rusting is hindered but if it does not adhere, the steel is easily attacked.

\*CARLE R. HAYWARD.

The Elastic Limit of Bronzes as Indicated by the Henning Recorder. E. S. SPERRY. Brass World, 7, 269-74.—An attempt to improve upon the detn. by using the Henning continuous automatic recorder in producing a magnified record, in conjunction with

a Riehlé testing machine. The detn. is of little importance compared to the other physical properties.

ROBERT KANN.

Segregation in Soft Steel Ingots. F. Wüst and H. L. Felser. Iron Age, 86, 730-34; see C. A., 5, 854.

L. A. Touzalin.

Chemistry of Iron Concrete (DONATH). 20.

Electrical Testing Machine (KAPP). 1.

Metallic Coatings by Friction (HILDEBRAND). 4.

Treatment of Anode Sludge (KERN). 4.

Ferromagnetic Manganese-Aluminium Bronzes (STEINER). 2.

- U. S., 999,814, Aug. 8. R. LUCKENBACH, Colwyn, Pa. Concentrating amalgamator.
- U. S., 1,000,217, Aug. 8. W. R. WALKER and J. H. GRAY, New York, N. Y. Manufacture of steel by subjecting the charge of an open-hearth furnace to the oxidizing action of flame and gases to remove the C, Si and P, removing the slag near the end of the heat, supplying a fresh basic slag to the charge in the same furnace, excluding the flame and gases from the furnace, and applying elec. heat to the charge in a non-oxidizing atm. to deoxidize and desulfurize the metal of the bath.
- U. S., 1,000,321, Aug. 8. I. L. Briggs, Clarksburg, W. Va. Assignor to Lillie Pearlman, Philadelphia, Pa. Rotary reducing and refining furnace having a cylindrical main portion, annular circumferential chambers at the ends of and communicating with the main portion and forming diametrical enlargements of the furnace, one of the chambers being of greater diam. than the other, the furnace having openings at its ends, the opening at the end having the greater enlargement being larger than the opening at the other end and the latter leading to the intake of a stack.
- U. S., 1,000,328, Aug. 8. D. C. Daniels, Worcester, Mass. Utilizing the heat of hot worked metal products by passing the articles successively through a body of H<sub>2</sub>O in a closed chamber to generate steam and afterward through another body of H<sub>2</sub>O to heat the H<sub>2</sub>O and further cool the articles.
  - U. S., 1,000,579, Aug. 15. W. A. BUTCHART, Los Angeles, Cal. Ore concentrator.
- U. S., 1,000,856, Aug. 15. B. W. TRAYLOR, New York, N. Y. Continuous filter for pulp, slimes, etc., comprizing a tank and a traveling series of flexibly connected filter pans with suction and air pressure connections to the pans and a supporting wheel at each end of the course of the filter pans for directing them through the tank.

## 10. ORGANIC CHEMISTRY.

### J. BISHOP TINGLE.

Practical Organic Methods. P. Blumberg. Fortschritte Chem., 4, 177-94.—Review for the year ending Apr., 1911, under the headings: oxidation; reduction; halogenation; nitration; sulfonation; acetylation; formation of esters and ethers; alkylation; condensation.

J. J.

Annual Review of Organic Chemistry. A. MAILHE. Rev. gen. sci., 22, 689-98.

Nitrites of Alkylammonium Bases: Ethylammonium Nitrite, Dimethylammonium Mitrite and Trimethylammonium Nitrite. P. C. RAY AND J. N. RAKSHIT. Presidency Coll., Calcutta. Pres. Chem. Soc., 27, 71; J. Chem. Soc., 99, 1470-5.—The salts were

prepared by the interaction of AgNO<sub>2</sub> and the corresponding chlorides of the alkylammonium bases, by evapn. of the filtrate in a vacuum over H<sub>2</sub>SO<sub>4</sub>. Double decomp. of HgNO<sub>2</sub> and the free base in aq. soln. also gives these salts. Ethylammonium nitrite, NH<sub>2</sub>EtNO<sub>2</sub>, viscid liquid of a pale yellow-brownish color. It slowly decomps. in a vacuum even at ordinary temp. Dimethylammonium nitrite, NH<sub>2</sub>Me<sub>2</sub>NO<sub>2</sub>, is a limpid pale yellow liquid. Trimethylammonium nitrite, NHMe<sub>2</sub>NO<sub>2</sub>, is a pale green cryst. solid.

E. E. GORSLINE.

Nitrites of the Benzylammonium Series. Benzylammonium Nitrite and Dibenzylammonium Nitrite and their Sublimation and Decomposition by Heat. P. C. PAY AND R. L. DATTA. Proc. Chem. Soc., 27, 127; J. Chem. Soc., 99, 1475-7.—The nitrites were obtained by the interaction of AgNO<sub>2</sub> and mono- and dibenzylamine hydrochlorides resp.; the filtrates being evapd. in a vacuum. Benzylammonium nitrite, C<sub>7</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub>, when heated in a Sprengel vacuum commenced to sublime at 80°, giving white stellar clusters of crystals. At higher temp. the salt decomps. into N and benzyl alc. Dibenzylammonium nitrite, C<sub>14</sub>H<sub>16</sub>O<sub>2</sub>N<sub>2</sub>, sublimes at 110°, white scales. It m. and sublimes at 110° but on maintaining this temp. decomps. into NO, dibenzylamine and dibenzylamine nitrate.

E. E. G.

Triketohydrindene Hydrate. V. The Analogs of Uramil and Purpuric Acid. S. RUHEMANN. Cambridge. J. Chem. Soc., 99, 1486-92.—1,3-Diketohydrindamine, CaH4: (CO)2: CHNH2, was made by gradually adding powdered oximino-1,3-diketohydrindene (5 g.) to SnCl, (15 g.), dissolved in conc. HCl. The solid collected after 1 hr. was dried in a vacuum and decompd. by H2S; yellow crystals. The product was not entirely pure. 1,3-Diketo-2-benzylidenehydrindamine, CaH4: (CO),CHN: CHPh, was obtained by the action of BzH on the above amine, orange needles, m. 196° (decomp.). 1,3-Diketo-2-salicylidenehydrindamine, C16H11O3N, orange crystals, m. 248-9° (decomp.). 1,3-Diketo-2-anisylidenehydrindamine, C17H11O1N, orange needles from EtOH, m. 315-6° (decomp.). 1,3-Diketo-2-p-dimethylaminobenzylidenehydrindamine, C.H.: (CO)<sub>2</sub>: CHN: CHC<sub>5</sub>H<sub>4</sub>NMe<sub>2</sub>, red needles from EtOH and glac. AcOH, m. 239° (decomp.). Diketohydrindylidenediketohydrindamine, C<sub>6</sub>H<sub>4</sub>: (CO)<sub>2</sub>: CHN: C: (CO)<sub>2</sub>: C<sub>6</sub>H<sub>4</sub>, is obtained as its NH, salt by the oxidation of diketohydrindamine or by the action of (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> on hydrindantin, using Piloty and Finckh's directions (Ann., 333, 27 (1904)); red powder. It gives a blue soln. of the NH4 salt when dissolved in NH<sub>4</sub>OH and this reaction is so sensitive that it can be used to test distilled H<sub>2</sub>O for NH<sub>2</sub>. Diketohydrindylideneuramil, CO: (NHCO): CHN: C: (CO)<sub>2</sub>: C<sub>2</sub>H<sub>4</sub>, is prepared by adding uramil to dil. KOH after displacing the air by H and pouring the soln., gradually formed on warming, into a soln. of triketohydrindene hydrate. From the reddish purple soln. small brown needles separate; the K salt of the compd. On adding dil. HCl, uramil separates.

Action of Ammonia and Amines on 2-Phenyl-1,3-benzoxazine-4-one. A. W. TITHERLEY AND E. C. HUGHES. Univ. Liverpool. Proc. Chem. Soc., 27, 190; J. Chem. Soc., 99, 1493-1510.—Salicylbenzamidine, HOC<sub>6</sub>H<sub>4</sub>CON: CPhNH<sub>2</sub> can be prepared (1) by dissolving 2-phenyl-1,3-benzoxazine-4-one in EtOH, satg. with NH<sub>3</sub> and allowing the soln. to evap.; or by adding satd. aq. NH<sub>3</sub> to the soln., when the product crystallizes out after 0.5 hr. (2) By mixing Ph salicylate to freshly prepared benzamidine and after the deposition of the white crystals, adding EtOH and heating. From the yellow soln. colorless needles of o-hydroxytriphenylcyanidine ppt., m. 248°, and on careful dilution of the alc. filtrate the salicylbenzamidine crysts.; orange needles from C<sub>8</sub>H<sub>6</sub>-light petroleum, m. 136-7°. Hydrochloride, colorless needles from an acetone soln. of salicylamidine treated with conc. HCl, partly m. at 120°, losing NH<sub>2</sub>Cl and yielding 2-Ph-1,3-benzoxazine-4-one. Salicylethylbenzamidine, HOC<sub>8</sub>H<sub>4</sub>CON: CPhN-HEt, results from treating an alc. soln. of 2-Ph-1,3-benzoxazine-4-one with 33% EtNH<sub>4</sub>;

yellow needles from light petroleum, m. 118-9°. It gives a deep red color with FeCl<sub>2</sub>. When NHMe<sub>2</sub> is employed under various conditions, viscid, yellow syrups result. Small quants. of crystals were sometimes obtained, m. 90°-106°. When NHEt<sub>2</sub> is employed, a yellow syrup results which sets to a cryst. mass. On adding cold 5% NaOH and recrystg. the remaining solid, by dissolving in EtOH at 40°, and carefully adding H<sub>2</sub>O, 2-diethylamino-2-phenyldihydro-1,3-benzozazine-4-one (I) results; color-

less needles, m. 81°. Even in a solid state this compd. undergoes partial rearrangement into the open chain isomeride, which can be traced by the appearance of a yellow color. A soln. in cold C<sub>a</sub>H<sub>a</sub>, CHCl<sub>a</sub> or acetone upon evapn., leaves a syrupy mixt. of the 2 isomerides. On dissolving the base in cold dil. HCl to a colorless soln., a white microcrystallin ppt. of the hydrochloride results if the soln. is not too dilute. On warming this soln. with aq. NH, a yellow soln. results, from which salicylbenzamidine crysts. Diphenylamine and 2-Ph-1,3-benzoxazine-4-one in dry C<sub>2</sub>H<sub>4</sub> soln. yields salicyldiphenylbenzamidine, HOC, H, CON: CPhNPh; bright yellow powder from C, H, m. 92°. This compd. gives no reaction with FeCl, and is insol. in NaOH. With piperidine a yellow syrup resulted, apparently a mixt. of the open-chain and cyclic isomerides. o-Methoxybenzoylbenzamidine, C18H14O2N2, was prepared from benzamidine and Ph o-methoxybenzoate in alc. soln.; colorless needles from EtOH, m. 128°. It dissolves in dil. HCi, decompg. slowly at 15°, but at 50° deposits N-benzoyl-o-methoxybenzamide in 0.5 hr. Phenyl o-methoxybenzoate, six-sided prisms, m. 59° was prepared from o-methoxybenzamide which is best made by dissolving salicylamide in EtQH, adding a soln. of Na in EtOH and then MeI. By condensing 2-Ph-1,3-benzoxazine-4one, or salicylbenzamidine with benzamidine, o-hydroxytriphenylcyanidine (II) results, already described under another constitutional formula by Pinner (Ber., 23, 2934, 3824 (1890)). It is a pale yellow, microcryst. ppt., m. 245°. It does not show normal phenolic properties. The velocity of hydrolysis of o-methoxybenzoylbenzamidine is normal as compared with similar amidines, while that of salicylbenzamidine and its derivs. is abnormally high. E. E. G.

Optically Active Derivatives of 1-Methylcyclohexylidene-4-acetic Acid. W. H. PERKIN, JR AND W. J. POPR. Cambridge, Manchester. J. Chem. Soc., 99, 1510-29.

—1-Methylcyclohexylidene-4-acetic acid (I) (C. A., 2, 2783) could be resolved into

optically active isomers (Perkin, Pope and Wallach, C. A., 4, 1019) in spite of the fact that it contains no asymmetric C at. Another enantiomorphous compd. of this type has been found in the oxime of cyclohexanone-4-carboxylic acid (II) (Mills and Bain, C. A., 5, 479). The authors propose to call compds. of this type centroasymmetric. When dl-1-methylcyclohexylidene-4-acetic acid is reduced with Pd and H, 1-methylcyclohexyl-4-acetic acid, an inactive compd., results. A similar reduction of the d- or the l-isomeride yields the same compd. Likewise the treatment of the dl-acid or the active isomers with HBr yields 4-bromo-1-methylcyclohexyl-4-acetic acid in each case. By decompg. 4-bromo-1-methylcyclohexyl-4-acetic acid with Na<sub>2</sub>CO<sub>3</sub>, 4-methylene-1-methylcyclohexane results. l-1-Methylcyclohexylidene-4-acetic acid can be converted into 1-Me-Δ<sup>3</sup>-cyclohexene-4-acetic acid (Markwald and Meth, Ber., 39, 1174 (1906)), an inactive acid, and this compd. by loss of H<sub>2</sub>O gives dl-1-Me-Δ<sup>3</sup>-cyclohexene-4-

acetic acid, an externally compensated compd. When dl-1-methylcyclohexylidene-4-acetic acid is dissolved in a little dry CHCl<sub>2</sub>, cooled to —10° and treated with Br, the product obtained by evapg. the product and drying on a porous plate, consists of 2 isomers:  $\beta$ -4-dibromo-dl-1-methylcyclohexyl-4-acetic acid,  $C_2H_{14}O_2Br_2$ , needles from light petroleum, m. 145-6°;  $\alpha$ -dl-1-methyl-4-dibromocyclohexyl-4-acetic acid, more sol. in petroleum ether, b. 70-80°, needles, m. 106°. When the corresponding d- and l-acids are treated with Br, 2 optically active isomeric dibromides are obtained in each case. The  $\beta$ -dibromides are less sol. in petroleum ether than the  $\alpha$ -isomers and cryst. on slow evapn. of their solns. in EtOAc. l- and d, $\beta$ -Dibromo-1-methylcyclohexyl-4-acetic acids (III), prisms from light petroleum, m. 154°;  $[\alpha]_D$  — and + 2.3° in AcOEt

soln. I- and  $d_i\alpha$ -dibromides, needles from light petroleum, m. 102°. On crystg. together equal quants, of the d- and the  $l,\alpha$ -dibromides the product m. 105-6°, the m. p. of the externally compensated compd. When the externally compensated form of either  $\alpha$ - or  $\beta$ ,4-dibromo-1-methylcyclohexylidene-4-acetic acid is slightly warmed in Na<sub>2</sub>CO<sub>2</sub> soln. the product is externally compensated. 1-Methyl-4-bromomethylenecyclohexane (IV), b, 112-3°. On treatment with fuming HBr this compd. yields 4-bromo-4-bromomethyl-1-methylcyclohexane, CHMe: (CH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>: CBrCH<sub>2</sub>Br, b<sub>r-6</sub> 120- $5^{\circ}$  with some decomp. The  $\alpha$ - and  $\beta$ -isomerides, either of d- or l, 1-methylcyclohexylidene-4-acetic acid, yield the same product on treatment with Na<sub>2</sub>CO<sub>2</sub>; b<sub>40</sub> 112-13°. When pure  $l,\alpha,4$ -dibromo-1-methylcyclohexyl-4-acetic acid is mixed with 10 parts of 50% KOH to a paste and heated to b. p., an extraction of the acidulated product yields 1,1-methylcyclohexylidene-4-bromoacetic acid, MeHC: (CH,CH,), : C: CBrCO,H, m. 50°  $[a]_b$  —10.4 in EtOH soln. When the corresponding  $d_b\beta$ -dibromo-acid is used the d-isomer results. Pure 1,1-methylcyclohexylidene-4-bromoacetic acid, when treated with Cl at -5°, protected from light, yields 1,4-chloro-1-methylcyclohexyl-4chlorobromoacetic acid (V), a viscid syrup, feebly l-rotatory. When d,1-methyl-4-

bromomethylenecylcohexane is used there results, d,4-chloro-1-methyl-4-chlorobromo-methylcyclohexane (VI), oil, b<sub>26</sub> 171-3°. E. E. G.

Some Reactions of Gum Kino. J. L. SIMONSEN. Presidency Coll., Madras. J. Chem. Soc., 99, 1530-5.—When gum kino, obtained from Sterocarpus marsupium is methylated, there results, kino methyl ether, m. above 300°. It is an amorphous powder from alc.-H<sub>2</sub>O, and its mol. wt. indicates a formula C<sub>10</sub>H<sub>14</sub>O<sub>4</sub>(OMe)<sub>20</sub> in naphthalene. As it probably exists as a colloid in soln., however, the accuracy of this result is questionable. It is insol. in alkalies and dissolves in conc. H<sub>2</sub>SO<sub>40</sub> giving a purple-red soln. Its alc. soln. gives no coloration with FeCl<sub>2</sub>. Acstylbino is a pale brown powder from AcOH-H<sub>2</sub>O. When b. with dil. HCl, kino dissolves and a red gelatinous mass of kino red shortly forms. No trace of kinoin (Etti, Ber., 11, 1879 (1878)) could be detected and sugar was not present. When fused with KOH at 190-200°, catechol was found but no phloroglucinol could be detected. When oxidized with KMnO<sub>4</sub> in Na<sub>2</sub>CO<sub>3</sub> soln., kino methyl ether yielded veratric acid. When either kino or kino-red is oxidized with dil. HNO<sub>30</sub> ammonium quadroxalate is found.

E. B. G.

Synthesis of Derivatives of Thioxanthone. III. 1,4-Dihydroxythioxanthone. H T. Clarke and S. Smiles. Univ. Coll., London. J. Chem. Soc., 99, 1535-9.—

2'-Carboxy-2,5-dihydroxydiphenyl sulfide, C<sub>2</sub>H<sub>2</sub>(OH)<sub>2</sub>SC<sub>2</sub>H<sub>4</sub>CO<sub>2</sub>H, results from mixing together p-benzoquinone in AcOH and a paste of o-thiolbenzoic acid with AcOH, allowing to stand for 24 hrs. and treating the resulting o-dithiolbenzoic acid with H<sub>2</sub>SO<sub>3</sub>; prisms from H<sub>2</sub>O, m. 199°. 2'-Carboxy-2,5-dimethoxydiphenyl sulfide, C<sub>16</sub>H<sub>14</sub>O<sub>4</sub>S, prisms from EtOH, m. 195°. By dissolving the carboxydihydroxydiphenyl sulfide in H<sub>2</sub>SO<sub>4</sub>

results; deep orange prisms from EtOH, m. 289°. The alk salts are crimson but could not be obtained pure owing to the ease of oxidation. 1,4-Dimethoxythioxanthone, C<sub>10</sub>H<sub>11</sub>O<sub>2</sub>S, results (1) from treating the dimethoxy deriv. with H<sub>2</sub>SO<sub>4</sub>, (2) from the condensation of o-dithiolbenzoic acid and quinol dimethyl ether; orange prisms, m. 195°. In H<sub>2</sub>SO<sub>4</sub> soln. a red color results.

E. E. G.

Contributions to the Chemistry of the Terpenes. IX. Oxidation of Camphene with Hydrogen Peroxide. G. G. HENDERSON AND M. M. J. SUTHERLAND. Glasgow and W. Scotland Tech. Coll. J. Chem. Soc., 99, 1539-49.—Camphene was oxidized by dissolving in 4 times its wt. of glac. AcOH, adding 30% H<sub>2</sub>O, and b. at 60°. The chief ingredient of the product was a new acid, camphylic acid, C. H., CO. H., crystg. from H<sub>2</sub>O and light petroleum, m. 95°. There were also small quants. of 2 other acids present, 1 m. at about 70°, the other an oily liquid, b<sub>20</sub> 153°. Sodium camphylate crysts. from H<sub>2</sub>O in prisms. Ammonium salt, cryst. powder. Silver salt, finely cryst. powder. Chloride, b,, 110°. Bromocamphylic acid, C,H,,BrCO,H, lustrous prisms from light petroleum, m. 210°. Hydroxycamphylic acid, C10H16O3, prisms from light petroleum, m. 245°. Among the neutral products of the oxidation the largest in quant. was camphenilone, C.H.,O. Some isocamphenilanaldehyde, C., H., CHO, was separated as a bisulfite compd. in small pearly leaflets. The aldehyde forms waxy crystals, m. 69-70°. On exposure to air the aldehyde gives isocamphenilic acid. Semicarbasone of the aldehyde, prisms from MeOH, m. 191-2°. Along with the camphenilone a compd. was obtained, b<sub>14</sub> 94-6°. On treatment with phthalic anhydride the acid phthalate of an alc., CoH100, resulted, lustrous prisms from Et.O light petroleum, m. 163-4°. The alcohol, C.H.O, a waxy cryst. compd., m. 94-5°. Small quants. of camphene glycol and of a cryst. compd. m. 69° were also obtained.

Constitution of the Organic Ferrocyanides. E. G. J. HARTLEY. Foxcombe Lab. J. Chem. Soc., 99, 1549-53.—0.5 g. tetramethyl ferrocyanide was gradually heated with 30 drops conc. H<sub>2</sub>SO<sub>4</sub> in a Pt crucible until white fumes appeared. It was kept at this temp. for several hrs. The Fe was almost entirely converted into Fe<sub>2</sub>SO<sub>3</sub> and when a little H<sub>2</sub>O was added, the soln. filtered, and the filtrate tested with PtCl<sub>4</sub>, NH<sub>4</sub> and NH<sub>2</sub>Me chloroplatinates were found. When the hexamethyl compd. was similarly treated the Fe was completely detached from the rest of the mol., but only MeNH<sub>2</sub> chloroplatinate was found. When the hexamethyl compd. was decompd. by 10% aq. NaOH the filtrate was also found to be free from the NH<sub>4</sub> salt. The author regards the following formulas as the best now available.

Tetramethyl ferrocyanide. Hexamethylferrocyanogen dihydrogen sulfate.

The views of Friedel (Compt. rend., 104, 995 (1897)), Browning (J. Chem. Soc., 77, 1238 (1900)) and Briggs (C. A., 3, 153; 5, 3018) were presented. The author favors the supposition that the ordinary structural theories of organic chemistry can at present be applied to this class of compds. more advantageously than Werner's so-called coordination theory or any allied system (see C. A., 4, 2293; 5, 75). E. E. G.

Alkaloids of the Bulbocapnine Group (Corydalis cava) and their Relation to One Another and to Apomorphine. J. Gadamer. Breslau. Chem. Ztg., 34, 1004-5; cf. C: A., 5, 1271, 1423, 2641.—Bulbocapnine, corytuberine and corydine contain the grouping C<sub>17</sub>H<sub>17</sub>N in common with apomorphine. On acetylation, bulbocapnine gave but 1 diacetyl derivative formed by rupture of the N ring. This, when methylated, gave bulbocapninemethine methyl ether (I). Bulbocapnine is (II) in which the posi-

tion of OMe and OH are probably but not certainly correct. When (I) is oxidized with alc., it gives dehydrobulbocapnine methyl ether iodide, which, on reduction, gave dl-bulbocapnine methyl ether, i. e., (II) with OMe instead of OH, which was sepd. into d- and l-forms with tartaric acid. By hydrolysis of the d- and l-compds., natural or d-bulbocapnine and l-bulbocapnine were obtained. The monobenzoylbulbocapnine behaves similarly. Corytuberine is the same as (II) except that I and 2 = OH, 3 and 4 = OMe. On treating with diazomethane besides N-methylated compds., 2 isomeric monomethyl ethers were obtained. Corydine is corytuberine methylated at (2) and isocorydine is methylated at (1).

E. J. WITZEMANN.

Some New Solvents for Cellulose and their Action on this Substance. Horace G. Deming. Univ. Wisconsin. J. Am. Chem. Soc., 33, 1515-25.—Conc. aqueous solns. of salts of strong acids and weak bases as ZnCl<sub>2</sub>, SbCl<sub>2</sub>, etc., dissolve cellulose (Schleicher and Schull filter paper no. 595) because of the presence of the free acid due to hydrolysis. Cellulose also dissolves in solns. of HgCl<sub>2</sub>, BiCl<sub>2</sub>, SnCl<sub>2</sub>, SbCl<sub>3</sub>, SnCl<sub>4</sub>, TiCl<sub>4</sub>, which contain a little HCl. The action of other chlorides, of some bromides and of the alkali and alkaline earth halides was also studied. A few of these salts in formic or trichloroacetic acids dissolve cellulose. When pptd. from these solns. the cellulose has great reducing properties and is easily hydrolyzed. A mixt. of 30 cc. of HCO<sub>2</sub>H (d. 1.22), 70 cc. HCl (d. 1.40) and 10 g. CaCl<sub>2</sub> readily dissolves cellulose; it may be recovered in a modified form by the sapon. of an ester by the HCl. Solns. of cellulose in HCO<sub>2</sub>H are rapidly hydrolyzed by HCl with the production of glucose, together with some furfural and AcOH. The theory of these phenomena is discussed.

Action of Substituted Hydrazines on  $\beta$ -o-Toluquinone. WILLIAM MCPHERSON AND CECIL BOORD. Ohio State Univ. J. Am. Chem. Soc., 33, 1525-31.— $\beta$ ,o-Toluquinone which is relatively stable, with unsym. benzoylphenylhydrazine in CHCl<sub>2</sub> yields ben-

zeneaso-m-cresol bensoate, formula (I), orange-yellow needles from ligroin, m. 98°. Attempts to isolate the intermediate compd. (II) were unsuccessful. When heated with KOH in Et<sub>2</sub>O, (I) is not changed, thus confirming its structure. On reduction with

Zn dust, (I) yields a compound, m. 159-60°, probably  $MeC_0H_3(OH)NHBz$ . Cold conc.  $H_2SO_4$  hydrolyzed (I), yielding benzeneazo-m-cresol; thin red plates from ligroin, m. 212°. Unsym. benzoyltolylhydrazine and  $\beta$ ,0-toluquinone yield tolylazo-m-cresol benzoate, light yellow-orange needles from ligroin, m. 93°. On hydrolysis it yields tolylazo-m-cresol, thin orange-red crystals from ligroin, m. 148°. S. J. B.

Hydantoins: The Reduction of Aldehyde Condensation Products of 1-Phenyl-2-thiohydantoin. TREAT B. JOHNSON AND CHARLES A. BRAUTLECHT. Sheffield Lab., Yale Univ. J. Am. Chem. Soc., 33, 1531-8.—1-Phenyl-2-thio-4-benzalhydantoin (C. A., 5, 2072, 2646) dissolves in Ba(OH), soln. and is pptd. by HCl. It is not changed by digestion with EtONa nor by b. dil. H<sub>2</sub>O<sub>2</sub>. On passing Cl into cold glacial AcOH in which the hydantoin was suspended, 4-(a-chlorobenzal)-1-phenyl-2-thiohydantoin, formula (I), resulted, colorless prisms from hot AcOH or alc., m. 236-7°. 4-(a-Bromo-

bensal)-1-phenyl-1,2-thiohydantoin, yellow plates from alc., m. 211°, yield 60%. 1-Phenyl-2-thio-4-benzalhydantoin is reduced practically quantitatively to 1-phenyl-2thio-4-benzylhydantoin with Na-Hg and slightly with Al-Hg. Attempts at reduction with FeSO, in NH, soln., with Zn dust and AcOH and with HI were unsuccessful. 1-Phenyl-4-benzalhydantoin (C. A., 5, 2646) on reduction with Na-Hg yields the hyacid PhNHCONHCH(CH,Ph)CO,H. 1-Phenyl-2-thio-4-benzylhydantoin dantoic and EtBr in presence of EtONa yield 1-phenyl-2-ethylmercapto-4-benzylhydantoin (II), viscous oil, which on digestion with HCl gave a quant. yield of 1-phenyl-4-benzylhydantoin. The latter with Ba(OH), gave phenylalanine, CO, and PhNH, 1-Phenyl-2-thio-4-benzylhydantoin was not hydrolyzed by b. Ba(OH), or NaOH. 1-Phenyl-2-thiohydantoin and di-Et oxalate (1 mol.) in presence of EtONa gave an 82% yield of 1-phenyl-2-thiohydantoin-4-glyoxylic acid, (III), pale yellow needles from alc., m. 240°. On reduction with Na-Hg, 1-phenyl-2-thio-4-anisalhydantoin gave a quant. yield of 1-phenyl-2-thio-4-(p-methoxybenzyl) hydantoin, slender colorless prisms from alc., m. 171°. By reduction of 1-phenyl-2-thio-4-piperonalhydantoin with Na-Hg, a 70% yield of 1-phenyl-2-thio-4-piperonylhydantoin was obtained, straw-colored needles from alc., m. 172-3°.

Condensation of p-Dibromobenzene with Xanthone: A Contribution to the Knowledge of Quinocarbonium Salts. L. H. Cone and C. J. West. Univ. Michigan. J. Am. Chem. Soc., 33, 1538-48.—p-Phenylenedixanthenol (C. A., 4, 1175), colorless crystals, m. 176-7° dissolved in  $C_6H_6$  and treated with HCl gas yields p-phenylenediquinoxanthenol chloride hydrochloride and p-phenylenedixanthenol chloride, formula (I), colorless crystals from  $C_6H_6$  and ligroin, darkens 210°, m. 259-60°. Mol. Ag

$$\begin{bmatrix} \bigcirc \begin{matrix} C_0H_4 \\ C_0H_4 \end{matrix} \\ CCl \end{bmatrix} \begin{matrix} C_0H_4 \end{matrix} \qquad \begin{matrix} C_0H_4OC_0H_4Br \cdot HBr \\ CC_0H_4Br \end{matrix} \qquad \bigcirc \begin{matrix} C_0H_4 \\ C_0H_4Br \end{matrix} \\ CII) \qquad \qquad (III)$$

removes the Cl from (I), forming an unsatd. hydrocarbon which could not be isolated but which is a true isolog of triphenylmethyl. Ferric chloride double salt of (I). Ca.Ha.O.Cl. (FeCl.), orange-red crystals. Zinc chloride double salt, red. Stannic chloride double salt, orange. Mercuric chloride double salt, dull red. Perbromide of (I), C2.H2.O2.Cl2Br4, finely divided yellow crystals. Periodide, nearly black. With HClO4 the original xanthenol yield the perchlorate, CaH2003(ClO4)2 reddish yellow crystals. Acid sulfate, C22H20O2(HSO4)2. p-Phenylenediquinoxanthenol bromide hydrobromide, reddish brown. p-Phenylenediquinonxanthenol bromide, red. Ferric chloride double salt with p-bromophenylxanthenol (C. A., 4, 1175) C10H112BrOCl.FeCl2, orange-yellow. Zinc chloride double salt, dark orange plates, m. 235° (not sharply). Stannic chloride double salt, dark yellow crystals, softens 250°, m. 257°. Perbromide of the chloride C1. H12 BrOCl. Br2, fine, light orange needles from CS2, m. 201-2°. Periodide, dark shining purple needles, m. 188°. Acid sulfate, C1. H12BrO.HSO4. H2SO4, large plated brown in transmitted light, greenish in reflected light, m. 77-8°. On adding AcBr to p-bromophenylxanthenol in CaHa, p-bromophenylquinoxanthenol bromide hydrobromide (II) is obtained as dark orange-red crystals. p-Bromophenylxanthenol bromide (III), colorless, becoming red in moist air. Zinc bromide double salt, C. H., BrOBr. ZnBr. bright red crystals, m. 250°. Mercuric bromide double salt, yellow crystals, m. 247-8°. Bromide perbromide, C<sub>10</sub>H<sub>12</sub>BrOBrBr<sub>2</sub>, m. 188°. Bromide periodide, purple, m. 211-2° STUART J. BATES.

Conversion of Benzenesulfonedibromoamide into Dibromobenzenesulfonamide by means of Concentrated Sulfuric Acid. J. H. Castle. Univ. Virginia. Am. Chem. J., 45, 219–23.—Corresponding to the conversion of tribromophenol bromide into tetrabromophenol by conc. H<sub>2</sub>SO<sub>4</sub> (Ber., 12, 1005) it is found that at room temp. conc. H<sub>2</sub>SO<sub>4</sub> instantly converts benzenesulfonedibromophenol into a dibromobenzenesulfonamide (probably the p-deriv.); a little Br is liberated at the same time and there is also formed a little of a white cryst. compd., m. 135–140°, which with alkali liberates NH<sub>4</sub>.

STUART J. BATES.

Reaction between Unsaturated Compounds and Organic Zinc Compounds. E. P. Kohler, G. L. Heritage and A. L. MacLeod. Am. Chem. J., 46, 217-36.-While Zn bromoacetate appears to form only 1,2-addition products with all kinds of unsatd. compds. (C. A., 4, 2270), the Zn derivs. of other a-bromo esters give both 1,2- and 1,4-addition products. The former appear as Zn compds. that give unsatd. OH esters when treated with an acid, the latter as Zn compds. that with an acid give satd. ketonic esters and as  $\delta$ -lactones. To ascertain the factors that influence the mode of addition, the reaction product was roughly analyzed by heating it with Na<sub>2</sub>CO<sub>2</sub> soln. Thus the unsatd. lactone and the ketonic ester give the ketonic acid, the OH ester being but slowly hydrolyzed; the wt. of this acid gives the amt. of the 1,4-product. The results with benzalacetophenone are: BrCH<sub>2</sub>CO<sub>2</sub>Me gives 100% 1,2-addition product, BrCHMeCO.Me gives 55% of 1,2- and 45% of 1,4. BrCHEtCO.Me gives 50% of 1,2- and 50% of 1,4-; both BrCMe<sub>2</sub>CO<sub>2</sub>Me and BrCH(CO<sub>2</sub>Me)<sub>2</sub> give 100% of 1,4-addition product. The procedure formerly used (C. A., 4, 2270) was followed except that in certain cases Cu acetoacetic ester was added as a catalyst. By heating the ester resulting from the reaction of bromoacetic ester and benzalacetophenone with Na<sub>2</sub>CO<sub>2</sub>, γ-benzal-β-hydroxy-β-phenylbutyric acid, PhCH: CHC(OH)PhCH<sub>2</sub>CO<sub>2</sub>H, colorless needles from Et<sub>2</sub>O, m. 147° is formed. When heated slowly it loses CO<sub>2</sub> and H<sub>2</sub>O below its m. p. Under certain conditions an oil was obtained as well as the ester of the above acid; with alc. KOH this oil yields  $\beta$ -phenylcinna mylideneacetic acid, PhCH: CHCPh: CHCO2H, slender, very pale yellow needles, m. 145-6°. From benzalacetophenone and Et bromopropionate were prepared: (a) Ethyl γ-benzal-β-phenyl-βhydroxy-a-methylbutyrate, PhCH: CHC(OH)PhCHMeCO, Et, alender needles from alc.

or Et<sub>4</sub>O m. 107°. (b) Ester, stereoisomer of (a) large transparent prisms, from alc. m. 81°. The product of the reaction when heated with Na,CO, yields (c) γ-benzoyl-βphenyl-a-methylbutyric acid. BzCH-CHPhCHMeCO.H. fine feathery needles from Et.O+ ligroin m. 149°. [In the original paper there is a misprint (c) being called  $\gamma$ -benzal, etc.—ABSTR.] Br does not combine with (c) nor does boiling alc. KOH decomp. it. (d) Acid, stereoisomer of (c) slender needles, m. 105°. It is probable that I or 2 lactones are formed at the time as (a), (b), (c) and (d). Methyl ester of (c), slender needles from MeOH, m. 68°. Ethyl ester of (c) needles m. 41°. Bromobutyric ester and benzalacetophenone with Cu acetoacetic ester as catalyst yield (a) γ-benzoyl-βphenyl-a-ethylbutyric acid, needles m. 181°. Methyl ester slender needles from MeOH, m. 95°. (b) Methyl γ-benzal-β-phenyl-β-hydroxy-a-ethylbutyrate, needles m. 117°. Et bromoisobutyrate and benzalacetophenone in conc. solns. yield mostly unsatd. lactone; in dil. solns. a ketonic ester, but if the latter soln. is b. a long time the yield of lactone is increased. The following compds. were obtained from the reaction product, (a) lactone, C<sub>10</sub>H<sub>18</sub>O<sub>2</sub>, colorless needles, m. 97°. It reduces a cold acetone soln. of KMnO, and combines with Br below oo. When b. with Na, CO, or alc. KOH it yields (b) γ-benzoyl-β-phenyl-a, α-dimethylbutyric acid, slender needles from MeOH, m. 159-60°. (c) Ethyl ester of (b) needles from MeOH, m. 83° was found in the original reaction product. Methyl ester of (b) needles from MeOH, m. 92°. On adding Br to a hot soln. of (b) in CHCl<sub>2</sub> or CCl<sub>4</sub> γ-bromo-γ-benzoyl-β-phenyl-α,α-dimethylbutyric acid, was obtained. It was purified by adding CCl, to its acetone soln. and then cooling in a freezing mixt.; long, colorless needles, m. 186° (decomp.). Methyl ester, large prisms, m. 125°. Ethyl ester, slender needles or thin plates, m. 131°. The bromo acid dissolves readily in dil. Na CO, soln. but slender needles soon deposit, which consist of a mixt. of the 2 stereoisomeric lactones, γ-benzoyl-β-phenyl-α,α-dimethyl-

PhCH.CHBz butyrolactons, | | O, both appear as colorless needles, m. 113° and 173° Me.C—CO—

resp. Both dissolve readily in alc. KOH; if the soln. is acidified after standing for some time they give only complex products, if acidified immediately they give  $\gamma$ -benzoyl- $\gamma$ -hydroxy- $\beta$ -phenyl- $\alpha$ ,  $\alpha$ -dimethylbutyric acid, needles from Et<sub>2</sub>O, which lose H<sub>2</sub>O on heating and m. 126°. When heated at 120° for several hrs. the acid passes completely into the lactone m. 113°. Br and Me malonate yield methyl bromomalonate, BrCH(CO<sub>2</sub>Me)<sub>2</sub>, colorless mobil liquid, b<sub>22</sub> 145°, this with benzalacetophenone and Zn yielded very little, if any, 1,2-addition product but gave methyl  $\gamma$ -benzoyl- $\beta$ -phenyl-ethylmalonate, large colorless needles from MeOH, m. 107°. With alc. KOH the ester gave a dibasic acid (Ann., 294, 332) which at 170° lost CO<sub>2</sub> and yielded  $\gamma$ -benzoyl- $\beta$ -phenylbutyric acid.

Anhydrous Formic Acid. James B. Garner, Blair Saxton and H. O. Parker. Peck Chem. Lab., Wabash Coll. Am. Chem. J., 46, 236–40.—HCO<sub>2</sub>H is best purified by repeated distillation over anhydrous CuSO<sub>4</sub>; m. 35°; b<sub>120</sub> 50°, b<sub>741</sub> 99.7°, d<sub>10</sub> 1.2322, d<sub>16</sub> 1.2260, d<sub>20</sub> 1.2200, d<sub>20</sub> 1.2139, d<sub>20</sub> 1.2078, d<sub>20</sub> 1.2019, d<sub>40</sub> 1.1957. Its viscosity varies from 0.0226 c. g. s. units at 10° to 0.01218 at 40°. Other physical constants are under investigation.

STUART J. BATES.

Group of Synthetic Organic Colloids. E. Wedekind. Strassburg. Z. Chem. Ind. Kolloide, 8, 303-4.—Several homologs of the general formula

have been prepared in the colloidal form by dissolving in AcOH, dil. with  $H_2O$  or triturating with alkali, then taking up with  $H_2O$ . The mechanism of the reactions depends on the amphoteric nature of the compd. forming a sol. salt with either the acid or alkali, then hydrolyzing to the colloidal form.

H. ISHAM.

Delépine's Phosphorescent Esters. JAIME F. HERNÁNDEZ AND ANGEL C. CERDAN. Labs. anál. quim. gen. quim. orgán. fac. cien., Univ. Central. Anales soc. españ. fis. quim., Q, 17-26.—Dimethyl dithiocarbonate was prepared as follows, with better yield than by the method described by Delépine. To a soln. of 20 g. KOH in 100 g. MeOH (kept cool by current of H<sub>2</sub>O) are added slowly, with constant stirring, 25 g. CS<sub>2</sub>; when this is dissolved, add slowly 40 g. Me<sub>2</sub>SO<sub>4</sub> and, when the reaction is complete, dil. with H<sub>2</sub>O until the white ppt. of MeKSO<sub>4</sub> is dissolved and di-Me dithiocarbonate seps. as a yellowish oily liquid. The latter is distilled with steam, sepd. from H<sub>2</sub>O and finally rectified and preserved over CaCl<sub>2</sub>. The white fumes observed by Delépine are not produced if the ester is pure and dry and are also absent in an atm. of moist CO<sub>2</sub>; these fumes are not phosphorescent and are posterior to the chemical action. Phosphorescence is greatly intensified if a solid object be interposed in the vapor of the ester carried from a tube by a rapid current of air; the same thing happens when the ester is rubbed between the hands, or in a mortar, or when a vessel containing it is moved. The phenomenon is probably a case of triboluminescence influenced by moisture and pressure. In presence of moisture the above ester is probably hydrolyzed with liberation of Me dithiocarbonate which is oxidized to S: C(OMe)SSC(OMe): S. In such a case small proportions of the 2 latter compds, would act as active substances and the unchanged vapor of the ester as the diluent. The white fumes are soluble in alc., insol. in H<sub>2</sub>O, and contain H<sub>2</sub>SO<sub>4</sub> and a substance which reduces KMnO<sub>4</sub> and AgNO<sub>2</sub>. The above phenomenon is very similar to the phosphorescence of P in the effect of moisture and pressure, the occurrence of intermittent luminescence, and the influence of petroleum, CaHa, etc.; likewise it was found, from the rate of discharge of a Curie electroscope, that air is ionized by the phosphorescence of this ester. No ultraviolet radiation could be detected by comparison of glass and quartz over a photographic plate, but radiations capable of passing through wood and affecting a photographic plate were detected after an exposure of 17 days, the plate being 2 cm. from the surface of the ester. H. S. PAINE.

Anthesterol. T. Klobb. Ann. chim. phys., 24, 134-44; cf. C. A., 3, 2146.— Anthesterol, m.  $185-95^{\circ}$ ,  $[\alpha]_0$  69.3° (2.5% CHCl<sub>2</sub> soln.), has the comp.  $C_{21}H_{12}O$ .3 $H_2O$ , not  $C_{28}H_{48}O$  as previously given. It loses its  $H_2O$  at 100° and then has  $[\alpha]_D$  75.4°. With Ac<sub>2</sub>O at 145° it forms a product  $[\alpha]_b$  83°, which can be resolved into 3 fractions: (1)  $\alpha$ -Acetate,  $C_nH_nOAc$ , hexagonal lamellas, m. 240-5°,  $[\alpha]_0$  91.2°, very slightly sol. in alc. (2)  $\beta$ -Acetate, pearly lamellas with truncated prisms, m. 220-7°,  $[\alpha]_0$ 73.9°, somewhat more sol. than the  $\alpha$ -form. (3) A fraction m. 185-95°,  $[\alpha]_D$  71.7°, more sol. than either (1) or (2) and probably consisting of a mixt. of these compds. Alc. KOH hydrolyzes the α-form to α-anthesterol, m. 220-2°, while Br gives 2 isomeric bromo derivatives, CnH50OAcBr; one, less sol., forms needles or hexagonal crystals grouped in rosets, m. about 185°, [a] 121-33° (3% C<sub>2</sub>H<sub>4</sub> soln.); the other, small needles or lamellas, m. 175-80°,  $[\alpha]_0$  58.8-63.9°, turns pinkish gray over H<sub>2</sub>SO<sub>4</sub>. The  $\beta$ -acetate gives with KOH a compd. showing the double m. p. of  $\beta$ -anthesterol, 160-5° and 195-8°, and with Br an amorphous dibromide, C<sub>11</sub>H<sub>49</sub>OAcBr<sub>2</sub>, m. 170-5°. The above facts indicate that anthesterol is a cholesteryl alc. decomp. by Ac<sub>2</sub>O into isomers corresponding to lupeol and amyrol on the one hand and the l-phytosterols on the other. CHAS. A. ROUILLER.

Crystallographic Study of Potassium p-Hydroxybenzoate. ARISTIDE ROSATI. Ist. min. r. univ. Roma. Atti accad. Lincei, 20, II, 53-4.—Comanducci's salt, p-

 $HOC_aH_aCO_aK.3H_aO$  (Gazz. chim. ital., 32, part I), forms transparent trimetric crystals with somewhat corroded or striated faces, a:b:c=0.6471:1:1.7936, (001), (010), (035), (065), (111), (885), tabular along (001), perfect cleavage along (001), imperfect along (010) and (100), plane of optical axes parallel to (010). C. A. R.

Syntheses of Pyrazolones from a  $\gamma$ -Pyrone Compound. F. Carlo Palazzo and Raffaele Liverani. Atti accad. Lincei, 20, II, 55–60.—From di-Et  $\alpha,\alpha'$ -dimethyl $\gamma$ -pyrone- $\beta,\beta'$ -dicarboxylate and N<sub>2</sub>H<sub>4</sub>.H<sub>2</sub>O were obtained 3 products: (1) Ethyl 3-methyl-5-pyrazolone-4-carboxylate, needles, m. 195–6°, gives an intense reddish violet color with FeCl<sub>2</sub> and is identical with the product obtained from N<sub>2</sub>H<sub>4</sub>.H<sub>2</sub>O and AcCH(CO<sub>2</sub>Et)<sub>3</sub>; yield, about 2%. (2) A compound isomeric with (1), softens 50–60°, m. 125–30°, gives a reddish brown color with FeCl<sub>2</sub>. (3) A compound which apparently has the comp. C<sub>11</sub>H<sub>16</sub>O<sub>4</sub>N<sub>4</sub>, m. very indefinitly about 143–5°, and gives an intensely reddish brown color with FeCl<sub>2</sub>. Chas. A. Rouiller.

Hydroxyhydroquinone Derivatives. V. G. BARGELLINI AND S. AURELI. Ist. chim. r. univ. Roma. Atti accad. Lincei, 20, II, 118-24.—In the presence of FeSO<sub>4</sub>, K<sub>x</sub>S<sub>2</sub>O<sub>8</sub> converts peonol into 4-methoxy-2,5-dihydroxyacetophenone, scales, m. 164°; diacetate, needles, m. 118-9°; dibenzoate, yellowish white, cryst. powder, m. 215°. With Me<sub>x</sub>SO<sub>4</sub> in the presence of NaOMe, the dihydroxy compd. gives, besides a little 2,4,5-(MeO)<sub>2</sub>C<sub>8</sub>H<sub>2</sub>Ac, 4,5-dimethoxy-2-hydroxyacetophenone, needles, m. 114-5°, gives a green color with aq. FeCl<sub>2</sub>; acetate, needles, m. 146-7°. 4,4',5'-Trimethoxy-2'-hydroxyacetophenone, from the hydroxyacetophenone and anisaldehyde, red needles, m. 130°, sol. in conc. H<sub>2</sub>SO<sub>4</sub> with intense orange-yellow color. HBr (d. 1.47) hydrolyzes 2,4,5-(MeO)<sub>2</sub>C<sub>8</sub>H<sub>2</sub>Ac to 5-methoxy-2,4-dihydroxyacetophenone, needles, m. 166°; acetate, needles, m. 127-8°.

Action of Aromatic Acetones on the Monosodium Derivative of Benzyl Cyanide. F. Bodroux. Fac. sci. Poitiers. Bull. soc. chim., 9, 758-61.—Ethylenic nitriles, RR'C: CPhCN, are formed from ketones RCOR' and PhCHNaCN only when the groups R and R' are cyclic. Thus, AcPh gives only free PhCH<sub>2</sub>CN, while BzPh (45.5 g.) yields 30 g. of 1,1,2-triphenylacrylonitrile, prisms, m. 166-7°, sol. in hot H<sub>2</sub>SO<sub>4</sub> with a violet color, hydrolyzed by NaOH in amyl alc. to the amide (Heyl and Meyer, Ber., 28, 1799). From 23 g. p-MeC<sub>4</sub>H<sub>4</sub>Bz is obtained 10 g. of 1,2-diphenyl-1-p-tolyl-acrylonitrile, long needles, m. 123°. 1,2-Diphenyl-1,α-naphthylacrylonitrile, prisms, m. 174-5°; yield, 1.5 g. from 18 g. α-C<sub>10</sub>H<sub>7</sub>Bz. Dibenzylideneacetone reacts only slightly and no definit product could be isolated.

Chas. A. Rouiller.

p-Hydroxyphenylglyoxylic, p-Hydroxyphenylacetic and p-Hydroxyphenylglycolic Acids. J. Aloy and Ch. Rabaut. Bull. soc. chim., 9, 762-4.—p-AcNHC<sub>6</sub>H<sub>4</sub>Ac is oxidized by alk. KMnO<sub>4</sub> to p-acetylaminophenylglyoxylic acid, AcNHC<sub>6</sub>H<sub>4</sub>COCO<sub>2</sub>H, slightly yellow crystals, m. 186-7°, sol. in alk. with yellow color. Silver salt, white crystals from H<sub>2</sub>O-EtOH. Phenylhydrazone, yellow ppt., m. 200-2° (decomp.). Dil. HCl hydrolyzes it to p-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>COCO<sub>2</sub>H (Böhringer, Chem. Zentr., 1901, I, 237) which, on diazotization, gives p-HOC<sub>6</sub>H<sub>4</sub>COCO<sub>2</sub>H. This last acid is reduced by boiling HI (b. 127°) or by Sn or Zn and HCl to p-HOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CO<sub>2</sub>H, which can also be obtained by the action of HI on p-MeOC<sub>6</sub>H<sub>4</sub>CH(OH)CO<sub>2</sub>H (cf. Czaplicki, Kostanecki and Lampe, C. A., 3, 1272).

Reduction in the Presence of Finely Divided Palladium. II. P. BRETEAU. Bull. soc. chim., 9, 764-70; cf. C. A., 5, 3397.—Pd sponge (7-8 g.) is dissolved in 20 cc. aqua regia, evapd. to dryness with conc. HCl until all the HNO<sub>3</sub> is removed, the PdCl<sub>2</sub> digested with 5 cc. conc. HCl for several hrs., 20 cc. com. HCHO added, the soln. filtered, conc. (filtered) NaOH added until the ppt. is finely pulverulent, the mixt. allowed to

stand 24 hrs., the ppt, washed until the wash liquid suddenly goes through colored a deep black, the washing suspended until the ppt, no longer decrepitates (0.5-1.0 hr.). and then the washing resumed until the ppt. is pure and the wash liquid passes through clear. When 5 g. of this Pd black are mixed with 10 g. of phenanthrene in 150 g. cyclohexane and purified H is passed into the mixt, until there is no further absorption, the phenanthrene is reduced exclusively to the tetrahydride. Zelinsky's method (Ber., 31, 3203) does not work with phenanthrene, but if the Zn is dissolved in HCl in the presence of a certain amt. of pptd. Pd, the hydrocarbon is reduced to the tetrahydride; to the Zn (100 g.) and phenanthrene (10 g.) in 250 g. of 95% alc. is added PdCl<sub>2</sub> (5-10 g. Pd) in HCl (d. 1.17) and more HCl slowly added until all the Zn is dissolved. The same reduction is effected by electrolyzing 2.5 g. phenanthrene in 140 cc. EtOH, 19 cc. H<sub>2</sub>O and 7 cc. H<sub>2</sub>SO<sub>4</sub> with an 8 v. and 10 amp. current. The anode is of Pb and the anode liquid 20% H2SO4, while the cathode, which is enclosed in a porous vessel, is prepared by depositing, on a Pt-Ir cylinder which has been roughened in a sand blast and is immersed in a H<sub>2</sub>SO<sub>4</sub> soln. of PdSO<sub>4</sub> (1.5% Pd), a coating of Pd sponge by means of a 4 v. and 8 amp. current. CHAS. A. ROUILLER.

Hydroxyindazoles. V. Constitution. P. FREUNDLER. Bull. soc. chim., 9, 773-4; cf. C. A., 5, 3397.—The correctness of the constitution already assigned to the chlorohydroxyindazoles obtained from o-substituted azo acids has been proved directly by the facts given below. 3,5,7-Trichlorophenylindazole, obtained by passing dry Cl into an AcOH soln. of phenylindazole on the H<sub>2</sub>O bath, needles, m. 172.5°, volatilizes without decomp. The same compd. is obtained by the action of PCl<sub>2</sub> in the presence of POCl<sub>2</sub> on the dichlorohydroxyindazole, showing that the latter really contains the indazole group. Oxidation of the trichloro compd. with CrO<sub>2</sub> in AcOH gives a product identical with that obtained from the hydroxy compd. and cold alk. KMnO<sub>4</sub>, HNO<sub>2</sub> or NaOCl, viz., benzeneazo-3,5-dichlorobenzoic acid, red prisms, m. 142.5-3.0°; the position of the Cl ats. is shown by reduction of the acid with Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> to 3,5-dichloro-2-aminobenzoic acid. The hydroxy compd. gives with BzCl dichlorophenylindazole, long needles, m. 203.5-4°; and with Me<sub>2</sub>SO<sub>4</sub> methoxydichlorophenylindazole, tabular prisms, m. 144-4.5°. Hydroxy-5-chlorophenylindazole is oxidized by HNO<sub>2</sub> to the benzeneazochlorobenzoic acid, m. 127°, already described.

CHAS. A. ROUILLER.

Action of Organic Acids on Sodium Formate. OBCHSNER DE CONINCE. Inst. chim. gen. Montpellier. Bull. sci. acad. roy. belg., 1911, 440-2, 590-1.—When mixts. of HCO<sub>2</sub>Na.H<sub>2</sub>O and malonic, succinic, tartaric, BzOH, o-, m- or p-HOC<sub>2</sub>H<sub>4</sub>CO<sub>2</sub>H, tannic, gallic, p-O<sub>2</sub>NC<sub>3</sub>H<sub>4</sub>CO<sub>2</sub>H or cinnamic acid are slowly heated, double decomptakes place to a greater or lesser extent and HCO<sub>2</sub>H distils over. C. A. R.

C<sub>3</sub>-Fluorobromo Derivatives. V. FRED. SWARTS. Lab. chim. gén. univ. Gand. Bull. sci. acad. roy. belg., 1911, 563-89; cf. Ibid., 33, 439; 34, 307; 1899, 357; 1901, 383.—unsym.-Tetrabromoethane, prepared by b. CHBr<sub>2</sub>CH<sub>2</sub>Br with KOAc and K<sub>2</sub>CO, in alc. and treating the resulting CH<sub>2</sub>: CBr<sub>2</sub> with Br, b<sub>16</sub> 112.5°. Unlike (CHBr<sub>2</sub>), and CHBr<sub>2</sub>CH<sub>2</sub>Br, CH<sub>2</sub>BrCBr<sub>6</sub> easily exchanges only 1 Br for F with SbF<sub>6</sub>. Thus 1,044 g. CH<sub>2</sub>BrCBr<sub>6</sub>, heated 12 hrs. at 135° with 180 g. SbF<sub>6</sub> (1/4 mol.), gave 680 g. of a liquid b. 161-6° and containing 463 g. CH<sub>2</sub>BrCFBr<sub>2</sub> and 217 g. CHBr: CBr<sub>3</sub>; 47 g. of CH<sub>2</sub>BrCF<sub>2</sub>Br, b. 92-3°; 71 g. of material b. above 166° from which were obtained CBr<sub>2</sub>: CBr<sub>3</sub>, m. 56°, and a product b. 206-9°, apparently CHBr<sub>2</sub>CFBr<sub>2</sub>; and 12 g. of a substance b. 143°. With 2/4 mol. SbF<sub>6</sub> the chief product is CH<sub>2</sub>BrCF<sub>2</sub>Br; the secondary products are trifluorobromoethane, b. 24-5° (also obtained by heating CH<sub>2</sub>BrCF<sub>2</sub>Br<sub>3</sub> and CBr<sub>2</sub>: CBr<sub>3</sub>. The most interesting facts observed are the forma-

tion of appreciable amts. of CHBr: CBr, and the absence of CBr,: CFBr and CH<sub>2</sub>: CFBr. unsym.-Tribromofluoroethane, CH<sub>2</sub>BrCFBr<sub>2</sub>, b. 162.7°, d<sub>17.8</sub> 2.6054, m<sub>17.4</sub> 1.50215; it reacts energetically with alc. KOH, giving sym.-dibromofluoroethylene, CHBr: CFBr, b. 88.8°, d<sub>17.4</sub> 2.2890, which is oxidized by O to CHBr<sub>2</sub>COF (cf. the formation of CHFBrCOBr from the isomeric compd. CBr,: CHF, Ibid., 1898, unsym.-Fluorobromoethylene (cf. Ibid., 1909, 709), when prepared by the reduction of pure CH\_BrCFBr, in alc. with Zn dust, b. 6.8°. wasym.-Dibromodifluoroethane, CH\_BrCF\_Br, b. 92.8°, d<sub>17'4</sub> 2.2423, n<sub>17'8</sub> 1.44815; b. with KOAc and K.CO, it yields unsym.-bromodifluoroethylene, CHBr: CF., b. 6.2°, de 1.82. Ethyl bromodifluoroethyl ether, EtOCF, CH, Br, obtained from CF, BrCH, Br and NaOEt, b. 114-5°; with HNO, it gives 2 mols. HF and CH,BrCO,H. Similarly the methyl trifluorobromoethyl ether previously described was shown to have the constitution MeOCF, CHFBr, for it gives 2 mols. HF and CHFBrCO, H. With Br CFBr: CHBr gives unsym.-tetrabromofluoroethane, CFBr, CHBr, b. 211° (slight decomp.), b<sub>24</sub> 106.4°, d<sub>17.6</sub> 2.9094. unsym.-Difluorotribromoethane, CF<sub>2</sub>BrCHBr<sub>2</sub>, from CF<sub>2</sub>: CHBr, b<sub>784</sub> 143.4-3.5°, d<sub>17'8</sub> 2.60769, n<sub>D</sub><sup>17'5</sup> 1.50215; with alc. KOH, it gives a small amt. of an ethylene compd. b. 70-80° and ethyl difluorodibromoethyl ether, EtOCF<sub>2</sub>CHBr<sub>2</sub>, b<sub>20</sub> 67.2°, b. 155°, d<sub>17-2</sub> 1.9158, forming CHBr<sub>2</sub>CO<sub>2</sub>H with HNO<sub>3</sub>. Even with KOAc and K2CO, the yield of unsym.-difluorodibromoethylene, CF,: CBr, is small; the compd. adds Br to form diffuorotetrabromoethane, CF, Br. CBr, m. 99°, b. 185°, but is quite volatil even at room temp. The trifluorobromoethane (see above) obtained by the action of AgF on CF, BrCH, Br (and in small amt. in the fluorination of CH, BrCBr,) is very stable towards HgO and alc. KOH, and only when heated at 150° with NaOMe is an ether, b. 45°, formed, which, with HNO, gives HF and CH.FCO.H. The constitutions of the ethane and the ether are therefore to be represented by the formulas CH,FCF,Br and CH,FCF,OMe, resp. The most striking fact brought out in this investigation is the resistance of the grouping CF, Br against further fluorination · 2 F ats. attached to 1 C at. even render a Br at. attached to an adjoining C at. resistant towards fluorinating agents. The same inertness of Br in the group CF,Br is manifested towards alc. KOH and NaOEt, and when it is attacked, the chief reaction is the formation of an ether, not an ethylene compd. CF, : CH, and CF, : CHBr do not oxidize in the air, while CBr,: CH, and CFBr: CHF do, the group: CFBr being the most sensitive to O. All these facts can be explained only by the theory of residual valencies or partial satn. of the ats. CHAS. A. ROUILLER.

Walden Inversion. VI. Transformations of  $\beta$ -Aminobutyric Acid. EMIL FISCHER AND HELMUTH SCHEIBLER. Chem. Inst., Univ. Berlin. Ann., 383, 337-63; see C. A., 5, 3282.—Me  $d_i l_i \beta$ -aminobutyrate is best prepared by heating crotonic acid (100 g.) with aq. NH<sub>a</sub>, saturated at the ordinary temp. (1 l.), at 130-40° (bath temp.), during 24 hrs. The product is evaporated repeatedly with H<sub>2</sub>O, then with aq. HCl and finally treated once or twice with MeOH and dry HCl, at the ordinary temp. The excess of MeOH is removed under reduced pressure and the ester liberated by means of NH<sub>2</sub>, in MeOH, below o°. Colorless liquid with a strong odor, b<sub>12</sub> 54-5°; d<sub>20</sub> 0.993. Yield, about 61%.  $d_1l_2\theta$ -Aminobutyric acid, m.  $187-8^\circ$ ,  $191-2^\circ$  (cor.). It is prepared by b. the ester during 4 hrs., with H<sub>2</sub>O (10 pts.). The acid dissolves CuO only very slowly, in which respect it differs from  $\alpha$ -amino acids; the Cu salt is best prepared from Cu(OAc), on the H<sub>2</sub>O bath. After drying in air during 5 hrs. it contains 3 H<sub>2</sub>O, after 4 dys., 2 H<sub>2</sub>O. β-Naphthylsulfonyl-d,l,β-aminobutyric acid, C<sub>10</sub>H<sub>2</sub>SO<sub>2</sub>NHCHMeCH<sub>2</sub>-CO<sub>2</sub>H, prisms from H<sub>2</sub>O, softens 163° (cor.), m. 166-7° (cor.). Its formation may be used for the qual. and quant. detection of  $d_i l_i \beta$ -aminobutyric acid. Dimethyl  $\beta$ -aminodibutyrate, C10H10O4N, is formed together with the preceding ester, especially by Curtius' method (J. prakt. Chem., [2] 70, 204 (1904)); liquid, bis 135°; bir 144-5°; die 1.044. The resolution of Me d.l. \( \text{\$\text{\$\sigma}\$-aminobutyrate is best accomplished by mixing it with d-camphorsulfonic acid and dry Et,O, crystals of the l-ester salt are deposited and are purified by means of MeOH + Et<sub>2</sub>O (1:3 vol.), at oo, the salts being finally decomp. by treatment with the calculated quantity of NH, in MeOH. Methyl LBaminobutyrate, liquid, b, 54-5°; [a]19 -6.97° (±0.02°). Methyl d, \beta-aminobutyrate is obtained from the mother liquor, as described above; its b. p. is identical with that of the *l*-compd.;  $[\alpha]_D^{20}$  8.91° (±0.02°). As is shown later, these esters both contained about 75% of d,l-ester, because the acids which they gave had  $[a]_b$  -7.9° and 10.1°, resp. The correct value for the d-ester is probably  $[a]_{n}^{20}$  31°.  $l_{n}^{20}$ -Aminobut ric acid is prepared from the soln, of the crude l-ester, by hydrolysis with H.O. as described above for the d.l-acid and is purified by systematic fractionation from anhydrous MeOH; well-developed, long, thick prisms, decomp, and evolves gas about 220°; in H<sub>2</sub>O,  $[\alpha]_D^{20}$  —35.2°  $(\pm 0.2^\circ)$ .  $d,\beta$ -Aminobutyric acid is prepared in a similar manner to the *l*-isomer, which it resembles closely; in H<sub>2</sub>O,  $[\alpha]_{p}^{20}$  35.3° (±0.2°); in IN aq. HCl,  $[\alpha]_{\mathbf{D}}^{20}$  29.7° (±0.4°); in IN aq. NaOH,  $[\alpha]_{\mathbf{D}}^{20}$  14.7° (±0.4°).  $l_{\mathbf{A}}\beta$ -Aminobutyric acid does not react so easily with HNO<sub>2</sub> (H<sub>2</sub>SO<sub>4</sub> + NaNO<sub>2</sub>) as does the  $\alpha$ -amino acid; the product consists of  $d,\beta$ -hydroxybutyric acid. Under similar conditions, Me  $l,\beta$ -aminobutyrate forms Me  $d,\beta$ -hydroxybutyrate and Me  $d,\beta$ -aminobutyrate gives Me  $l,\beta$ -hydroxybutyrate, which was isolated as the Na butyrate. In both cases some d,l-compd. was also formed. These results show that HNO, produces the same reaction (considered optically) with the optically active  $\beta$ -aminobutyric acids as it does with their esters. Under the ordinary pressure, at oo, NO + Cl, or Br, react only slowly with the  $\beta$ -aminobutyric acids, in solns of halogen acids. In this respect the  $\beta$ -amino acids differ markedly from the  $\alpha$ -amino compds. The reaction takes place fairly readily, however, in conc. HCl, with excess of NOCl, under pressure, at the ordinary temp., during 2-3 dys. The product was purified by soln. in petroleum ether and conversion into the Na salt. In this manner the  $d_i l_i \beta$ -amino acid gave d,l,β-chlorobutyric acid, C<sub>4</sub>H<sub>7</sub>O<sub>2</sub>Cl; b<sub>16</sub> 103-5°. It was not quite pure but it was identified by conversion into the hydroxy acid. The d- and l, \beta-aminobutyric acids gave the l- and  $d_i\beta$ -chlorobutyric acids, resp. They were purified by means of their Ag salts. The amino acids employed contained d,l-compd., but, correcting for this, the results for  $[\alpha]_D$  show that the action of NOCl involves but little inversion into the d,l-isomer. The best known method of preparing  $d\beta$ -chlorobutyric acid is as follows: Syrupy  $l_i\beta$ -hydroxybutyric acid, from diabetic urin is b. with anhydrous MeOH and HCl (1%) and the resulting ester, after distillation, is treated with PCl. at the temp. of ice + NaCl; the product is mixed with ice and extracted with EtCl, because the chloro ester is volatil with Et<sub>2</sub>O; b. 148-52°;  $[\alpha]_{D}^{20}$  22.6°, this value is probably too low. The ester is hydrolyzed by means of HCl (d. 1.19), at the ordinary temp., during 2 dys. The resulting  $d_{i}\beta$ -chlorobutyric acid is purified by cooling its ligroin soln. to the temp. of ice + NaCl; large prisms from ligroin, m. 43-4.5°; in H<sub>2</sub>O,  $[\alpha]_D^{20}$  49.8° (±0.2°); in toluene,  $[\alpha]_D^{20}$  46.6° (±0.2°); in 1 N aq. NaOH,  $[\alpha]_D^{20}$ 41.3° (±0.4°). Silver salt, slender, colorless needles; it is stable towards light. Yield, about 90%. Silver d,l,β-chlorobutyrate resembles the preceding compd. closely. These results show that a Walden inversion must take place at least once in the transforma-HNO.

tions  $d,\beta$ -aminobutyric acid  $\longrightarrow l,\beta$ -hydroxybutyric acid and  $d,\beta$ -aminobutyric NOCl  $H_4O$ 

acid  $\longrightarrow l,\beta$ -chlorobutyric acid  $\longrightarrow d,\beta$ -hydroxybutyric acid. J. B. Tingle.

Preparation of Amino Acids from Keto Acid Phenylhydrazones and Aluminium Amalgam and the Production of Optically Active 7-Aminovaleric Acid. EMIL FISCHER AND REINHART GROH. Chem. Inst., Univ. Berlin. Ass., 383, 363-72.—7-Amino-

valeric acid is best prepared by the following process: Levulic acid and PhN<sub>2</sub>H<sub>2</sub> are allowed to interact in aq. soln. and the crude phenylhydrazone (200 g.), after being washed with H<sub>2</sub>O, is dissolved in alc. (2 l.) and mixed with H<sub>2</sub>O (500 cc.) and Al turnings (70 g.), which have been amalgamated by means of HgCl, soln. The mixture is well shaken and allowed to react during 15-20 hrs. The product is centrifuged, the solid mixed with H<sub>2</sub>O and again centrifuged and the combined liquors, after being b. with animal charcoal, are conc. to 150 cc., under reduced pressure. The liquid is extracted with Et<sub>2</sub>O and the aq. soln. is mixed with alc. (4 vols.); on adding Et<sub>2</sub>O the amino acid is deposited in rods. Yield, 70 g. = 60%.  $\beta$ -Aminobutyric acid is prepared in a similar manner from crude acetoacetic ester phenylhydrazone, the Al-Hg being allowed to react first at o°, then at 10-20°, finally a second portion of metal is added and the reduction continued at the ordinary temp. Yield, 56%. Alanine is formed in the same manner from pyruvic acid phenylhydrazone. Yield, 55%. This method is, however, inferior to the ordinary process for the preparation of alanine. Benzoyl-7-aminovaleric acid is best obtained by the use of NaHCO<sub>2</sub>, in place of NaOH. Yield, 150% of the parent acid. It is resolved into optical isomers by means of quinine, in alc.; at o°, crystals of the salt of the Lacid deposit readily if the liquid be inoculated, otherwize the formation of crystals may require weeks. The salt is decomp. by NaOH, in alc. + H<sub>2</sub>O, followed by HCl. l,γ-Benzoylaminovaleric acid, C<sub>12</sub>H<sub>14</sub>O<sub>2</sub>N, long needles from H<sub>2</sub>O, m. 131° (133° cor.), previously softening; in alc.,  $[\alpha]_D^{20}$  —21.9° (±0.2°). It is less readily sol. in H<sub>2</sub>O than the d,l-acid. Yield, 70%. When hydrolyzed by b. in a quartz vessel, during 15 hrs., with aq. HCl (20%), it gives d, \( \text{7-amino-} \) valeric acid, C<sub>2</sub>H<sub>11</sub>O<sub>2</sub>N, which is purified by means of Ag<sub>2</sub>O; crystals from H<sub>2</sub>O + alc., m. about 209° (214° cor.) (gas evolution); in H<sub>2</sub>O,  $[\alpha]_{D}^{20}$  12.0° (±0.2°). Yield, 86%. It was reconverted into the Bz deriv. which had  $[\alpha]_{D}^{20}$  —21.7° (±0.4°), showing that the hydrolysis as above had not caused the formation of any d,l-amino acid. Quining d,r-bensoylaminovalerate is contained in the mother liquor from the l-salt and is purified by repeated recrystallization from  $H_2O$ . The resulting  $d_{ij}$ -benzoylaminovaleric acid, prepared in the same manner as the isomer, was not completely separated from d,l-acid; in abs. alc.,  $[\alpha]_D^{20}$  16.5°  $(\pm 0.2^\circ)$ . After hydrolysis it gave  $l_{ij}$ -aminovaleric acid having, in (H<sub>2</sub>O?),  $[\alpha]_{p}^{20}$  —10.7° (±0.2°). J. BISHOP TINGLE.

Influence of Substitution in the Case of Components of Binary Solutions in Equilibrium. V. Fluorene and Polynitrobenzenes. R. KREMANN. Chem. Inst., Univ. Graz. Monatsh., 32, 609-17; see Ibid., 27, 125 (1906).—The eutectic mixture of fluorene and o-dinitrobenzene m. 77°, that of m-dinitrobenzene, m. 54° and that of the p-compd., m. 90°. The mixtures contain 53, 42 and 72% of fluorene, resp. 1,2,4-Dinitrotoluene and fluorene give a eutectic mixture containing 38% of the hydrocarbon; m. 44°. The above nitro derivs. do not combine with fluorene. Picric acid and fluorene form a eutectic mixture containing 55.5% of fluorene; m. 80.5°. Picric acid and fluorene combine in equimol. proportion, the resulting compound m. 84°. It gives a eutectic mixture with picric acid; m. 80° and contains 34% of fluorene. Its eutectic mixture with fluorene m. 80.5° and contains 55.5% of fluorene. The eutectic mixture of fluorene and 1,3,5-trinitrobenzene m. 86° and contains 64% of fluorene. These substances also form a compound containing fluorene and trinitrobenzene in the ratio 2:3 mols., m. 105°. Its eutectic mixture with 1,3,5-trinitrobenzene m. 98.5° and contains 20% of fluorene. Its eutectic mixture with fluorene m. 86° and contains 64% of fluorene. Fluorene and "trinitrotoluene" combine in equimol. proportion; the resulting substance m. 85°. Its eutectic mixture with fluorene m. 78.5° with "trinitrotoluene" m. 72°. They contain 57.5 and 17% of fluorene, resp. Fluorene and 2,4-dinitrophenol give a eutectic mixture containing 52% of the former; m. 74°.

These results show that in its ability to form mol. compds. fluorene resembles C<sub>0</sub>H<sub>0</sub> and phenanthrene closely, but that it differs greatly from naphthalene.

J. BISHOP TINGLE.

New Method for the Formation of Glucuronic Acid. ADOLF JOLLES. Vienna. Monatsh., 32, 623-9.—Glucuronic acid is formed by either of the methods described below, but the second is preferable. Yield, in both cases poor. (1) An aq. (2%) soln. of pure dextrose (5 l.) is treated with H<sub>2</sub>O<sub>2</sub> (12 vol. %; 100 cc.) at 37°, during 144 hrs., the H<sub>2</sub>O<sub>2</sub> being renewed at the end of each 24 hrs. The soln. is finally neutralized, treated with Pt black, then with yeast, filtered, conc. under reduced pressure and mixed with aq. NH<sub>2</sub> and conc. Pb(OAc)<sub>2</sub> soln. The Pb salt is washed with H<sub>2</sub>O at 70-80°, decomp. by H<sub>2</sub>S and the filtrate conc. at 40°, under reduced pressure. (2) The liquid after oxidation and fermentation as above, is filtered, treated with basic Pb(OAc)<sub>2</sub> and then Pb(OAc)<sub>3</sub> is added until no further ppt. is formed. The salt is washed 5 times by decantation, the ppt. being allowed to settle each time during 2-4 days. The purified salt is decomp. in the manner described above. The glucuronic acid was identical by means of its p-bromophenylhydrazine deriv. and by its oxidation to saccharic acid. (Also in Biochem. Z., 34, 242-7.)

J. BISHOP TINGLE.

Synthesis of Linear Diphthaloyl Benzene. I. ERNST PHILIPPI. II chem. Lab., Univ. Vienna. Monatsh., 32, 631-5.—1,5-Dibenzoylbenzene-2,4-dicarboxylic acid, Bz<sub>2</sub>C<sub>6</sub>H<sub>2</sub>(CO<sub>2</sub>H)<sub>2</sub>, is prepared from pyromellitic acid (5 g.), C<sub>6</sub>H<sub>2</sub> (90 g.) and AlCl<sub>6</sub> (10 g.), at 53°; the crude product being treated with HCl and the solid extracted with b. H<sub>2</sub>O. Long plates from PhNO<sub>2</sub>, needles from glacial AcOH, m. 263-4° (darkens). 1,4-Dibenzoylbenzene-2,5-dicarboxylic acid is formed together with the preceding compd. and is contained in the residue remaining after treatment with H<sub>2</sub>O, as above. It. is extracted by aq. Na<sub>2</sub>CO<sub>3</sub>: long, lancet shaped plates from PhNO<sub>2</sub>, slender needles from glacial AcOH, softens above 295°, m. 307-9°. When distilled with CaO (18-22 mm.), in an atm. of H, it gives p-dibenzoylbenzene. Diphthaloylbenzene is prepared

from either of the above acids (0.6 g.) and conc. H<sub>2</sub>SO<sub>4</sub> (20 g.), at 92-4°, during about 5 hrs., the product being b. with H<sub>2</sub>O and then with dil. aq. Na<sub>2</sub>CO<sub>3</sub>; bundles of slender, yellow needles from PhNO<sub>3</sub>, or glacial AcOH, not m. 370°, sublimes at a higher temp. Yield, about 58% of the acid. With alkaline "hyposulfite" it gives a dark red vat dye which colors cotton green, changing quickly to dark blue and finally, in air, to rose-red.

J. BISHOP TINGLE.

Friedel-Crafts' Reaction. OTTOKAR HALLA. Chem. Lab., Imp. and Roy. Ger. Univ., Prague. Monatsh., 32, 637-40.—Xanthone fails to react with phthalic anhydride and AlCl<sub>2</sub>; in C<sub>8</sub>H<sub>6</sub>, the product consisted of "benzoyl" and "toluylbenzoic acids," the latter m. 140° not 146°, as stated by Friedel and Crafts. The production of "toluylbenzoic acid" is ascribed to the presence of toluene in the C<sub>8</sub>H<sub>6</sub>. Expts. with phthalic anhydride, AlCl<sub>2</sub> and C<sub>5</sub>H<sub>6</sub> containing 10, 5, 3, 1 and 0.5% of toluene, resp. showed that toluylbenzoic acid was always obtained, even in the presence of a large excess of C<sub>8</sub>H<sub>6</sub>. It must be a primary product because it is not formed from "benzoylbenzoic acid," AlCl<sub>2</sub>, toluene and C<sub>8</sub>H<sub>6</sub>, nor from AlCl<sub>3</sub>, C<sub>8</sub>H<sub>6</sub>, toluene and (A). The term (A) is used to designate the solid substance which results after removing the excess of C<sub>6</sub>H<sub>6</sub> from the product of the interaction of AlCl<sub>6</sub>, phthalic anhydride and C<sub>6</sub>H<sub>6</sub>. These facts do not accord with the conclusion of Heller and Schülke (C. A., 3, 3, 18), that the Friedel-Crafts reaction includes, as one of its stages, a combination with

C<sub>0</sub>H<sub>0</sub>. The resulting material may then have the C<sub>0</sub>H<sub>0</sub> displaced by some other complex, such as naphthalene, if it be present.

J. BISHOP TINGLE.

New Leucomaine. J. UBEDA Y CORREAL. 7th Intern. Congr. Appl. Chem., IV. A2, 112-3; through J. Chem. Soc., 100, I, 396.—The leucomaine was obtained by treating fresh beef (29 kg.) with aq. HCl, evaporating, extracting the dry residue with alc., pptg. with HgCl, and treatment with H<sub>2</sub>S. Hydrochloride, colorless crystals with angles of 31° 33' and 148° 27', resp. Chloroplatinate, red, acciular prisms with angles of 48° 49' and 131° 16', resp. After treatment with H<sub>2</sub>S the resulting clear soln. reacts with AuCl, KI, CdI, and picric acid.

J. BISHOP TINGLE.

Preparation of Purely Aromatic Telluronium Compounds with Similar Hydrocarbon Residues. Charles Lederer. Chem. Lab., Univ. Brussels. Ber., 44, 2287-92.-Triphenyltelluronium iodide, Ph.TeI, is prepared from TeCl, and PhMgBr, in Et.O, at oo, the solid product, after treatment with alc., being dissolved in H<sub>2</sub>O and the iodide pptd. with KI; small needles from H<sub>2</sub>O, softens 245°, m. 247-9°. Bromide, from the iodide and AgBr; microscopic, prismatic needles from H<sub>2</sub>O, m. 259-60°. Chloride, from the iodide and AgCl; long, slender needles from abs. alc. + Et<sub>2</sub>O, m. 244-5°. It gives ppts. with HgCl, AuCl, SnCl, picric acid, K,CrO, and H,PtCl. The chloride is decomp. by PhMgBr into diphenyl and diphenyl telluride. Tri-p-tolyltelluronium iodide, (MeC.H.).TeI, is prepared like the Ph compd.; bundles of microscopic, hexagonal plates from alc., m. 232-3° (decomp.). Bromide from the chloride and KBr, or from the iodide and AgBr; crystals from H<sub>2</sub>O, or from alc. + Et<sub>2</sub>O, m. 265-6° (decomp.). Chloride, from AgCl; long, interlaced needles, or small prisms from abs. alc. + abs. Et.O., m. 260-1°. It gives ppts. with HgCl., SnCl., AuCl., pieric acid and H.PtCl. Tri-o-tolyltelluronium iodide, prepared like the isomer; small, microscopic, quadratic rods from H<sub>2</sub>O, or from alc. + abs. Et<sub>2</sub>O, m. 195-6°. Picrate, sparingly sol. Bromide. J. BISHOP TINGLE.

Nitrose Compound of Hydroxyhydroquinyl Dimethyl Ether and its Derivatives. RUDOLF FABINYI AND TIBOR SZEKI. Chem. Lab., Roy. F. J. Univ., Kolozsyár. Ber., 44, 2293-8; see C. A., I, 311.-2,5-Dimethoxy-4-nitrosophenol, ONC<sub>2</sub>H<sub>2</sub>(OMe)<sub>2</sub>OH, or 2,5-dimethoxy-1,4-benzophenoneoxime, O: C<sub>2</sub>H<sub>2</sub>(OMe)<sub>2</sub>: NOH, exists in a brickred and in a lemon-yellow form, this latter is produced from the former by repeated crystn. from glacial AcOH, or by hydrolyzing the Ac deriv. With Me<sub>2</sub>SO<sub>4</sub> and aq. NaOH it gives 1,2,5-trimethoxy-4-nitrosobenzene, ONC,H,(OMe),; long, pale reddish yellow needles from alc., m. 191°. Yield, 40% of the phenol. 4-Amino-2,5-dimethoxyphenol, H<sub>2</sub>NC<sub>6</sub>H<sub>2</sub>(OMe)<sub>2</sub>OH, is best prepared from the nitroso compd. by the action of dil., aq. NH<sub>4</sub> + H<sub>2</sub>S; m. 157°. Acetyl derivative, from Ac<sub>2</sub>O; crystals from alc., or H<sub>2</sub>O, m. 180°. Diacetyl derivative, from Ac<sub>2</sub> + AcONa; crystals from alc., m. 190°. Dipropionyl derivative, prepared like the preceding compd.; crystals from alc., m. 131°. At 50°, HNO, (50%) oxidizes 4-amino-2,5-dimethoxyphenol to 2,5-dimethoxy-1,4-quinone. Ethyl [2,5-dimethoxy-4-hydroxyphenyl]aminoformate, (MeO)<sub>2</sub>-.C.H. (OH)NHCO, Et, is obtained from the aminophenol and Et chlorocarbonate; Instrous plates from C<sub>2</sub>H<sub>2</sub>, m. 143°. Sodium salt, white, interlaced needles from alc. Acetyl derivative, from Ac.O; white crystals from alc., m. 135°. 2,5-Dimethoxy-4carbethoxyaminophenoxyacetic acid, (MeO), C, H, (NHCO, Et) OCH, CO, H, from the preceding ester, EtONa and Et bromoacetate; crystals from alc., or H<sub>2</sub>O, m. 108°. Sodium salt, readily sol. in H<sub>4</sub>O. 2,5-Dimethoxy-4-acetylaminophenoxyacetic acid, (MeO)<sub>2</sub>-C.H.(NHAc)OCH,CO.H, is prepared from 2,5-dimethoxy-4-hydroxyacetanilide, EtONa, alc. and Et bromoacetate; white crystals from alc., m. 172°. Certain of the above compds. exhibit varying degrees of antipyretic action which, however, does not exceed that of phenacetine. J. BISHOP TINGLE.

Diphenyl Derivatives. FRITZ MAYER. Chem. Lab., Phys. Assoc. Acad., Frankfort a/M. Ber., 44, 2298-305.—2-Acetylaminobenzene-1,3-dicarboxylic acid, the prep. of which is described, when warmed with Ac<sub>2</sub>O, gives an anhydride,

HO<sub>3</sub>CC<sub>4</sub>H<sub>3</sub> | ; pale yellow crystals, decomp. 217-8°. It is hydrolyzed by H<sub>2</sub>O

to the acid. When b. with conc. H<sub>2</sub>SO<sub>4</sub> (30 cc.) and H<sub>2</sub>O (20 cc.), the acid (10 g.) is hydrolyzed to 2-aminoisophthalic acid, which was diazotized and converted into a mixture of 2-hydroxy- and 2-iodoisophthalic acids, these were esterified with MeOH + HCl, at the ordinary temp. and the hydroxy ester extracted with dil. aq., NaOH. Methyl 2-iodoisophthalate, long needles, m. 50°. Acid, slender, almost white needles from H<sub>2</sub>O, decomp. 205-22°. At 200°, "natur Cu C" converts the ester into tetramethyl diphenyl-2,6,2',6'-tetracarboxylate; white needles from MeOH, m. 125-6°. Acid, white, iridescent plates, not m. 350°. Chloride, from PCl, and POCl, at 170°; solid from C<sub>2</sub>H<sub>4</sub> + ligroin, m. 189-90°, previously softening. It could not be reduced, nor could it be converted into pyrene. 3-Methyl-2-iodobenzoic acid was prepared from the amino compd., as described above; needles from C<sub>2</sub>H<sub>a</sub>, or MeOH, m. 145-6°, previously softening. Methyl ester, oil, b. 280-90°. At 220°, with "natur Cu C," it gives a product which, after hydrolysis, consists of 2,2'-dimethyldiphenyl-6,6'-dicarboxylic acid, white crystals from glacial AcOH, m. 230°, previously softening. Yield, very poor. Methyl o-aminocinnamate is deposited in yellow, silky needles, m. 65°. It was converted into methyl o-iodocinnamate; oily. No di-Ph deriv. could be obtained from it by fusion with Cu. o-lodobenzylideneaniline is formed from o-iodobenzaldehyde and PhNH<sub>3</sub>; white needles from MeOH, m. 75-6°. At 210-20°, "natur Cu C" and o-iodobenzaldehyde give diphenyl-o,o'-dialdehyde which was not purified, but was converted directly into the dioxime; yellow needles from dil. AcOH, m. 175-6°. Diphenyl-2,2'-diacrylic acid is obtained from the preceding dialdehyde, Ac.O and AcONa, at 145°; white crystals from glacial AcOH, m. 286° (gas evolution). It could not be converted into pyrene, but aq. KMnO<sub>4</sub> (1%) and Na<sub>2</sub>CO<sub>2</sub> oxidize it to diphenic acid. I. BISHOP TINGLE.

Racemic Substances (KRUYT) (LINDEN) (LADENBURG). 2.
Activity of Catalyzers (POAL, KARL). 2.
Hydrocyanic Acid, Benzaldehyde Solution (WORTH). 17.
Methyl Guanidines (SCHENCE). 17.
Blood Pigments (PILOTY). 11.

## 11. BIOLOGICAL CHEMISTRY.

wm. j. gies. GENERAL.

The Importance of Two Electrostatic Factors in the Osmosis of Electrolytic Solutions. Normal Osmotic Movements. Pierre Girard. Compt. rend., 153, 401-4.—Osmosis of electrolytes is dependent upon the electrostatic condition of the ions. The direction of osmosis may be altered by changing the sign of the electrostatic condition. This would explain the osmosis of salts in vivo which often takes place in the sense opposit to that in vitro and generally ascribed to selective cell action.

G. M. MEYER.

### METHODS AND APPARATUS.

Iodometric Method for the Determination of Acid in the Gastric Juice, and its Clinical Advantage. M. Wezrumba. Med. Klin., Bern. Intern. Beitr. Path. Therap., 3, 53-85.—Greater amts. of acid were found in gastric juice when detd. by the iodometric method than by the Congo red titration. In pathological cases different amts. of acid are indicated by the 2 methods. The iodometric method consists essentially in adding to the liquid to be examined KI and KIO<sub>3</sub> solns., starch paste, and then titrating very carefully with 0.1 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

C. A. Brautlecht.

Colorimetric Determination of Sugar in the Blood. LEONHARD WACKER. Path. Inst., Univ. München. Zentr. ges. Physiol. Path. Stoffw., 6, 524-8.—A response to the communication of Forschbach and Severin (cf. C. A., 5, 1473, 3086). W. compares his method with that of F. and S. (cf. C. A., 5, 2392) and finds that the results do not agree. (This does not refer to the second article below.—Abstr.)

C. A. BRAUTLECHT.

A New Test for Sugar in Urin. L. DEJAGER. Leeuwarden, Holland. Zentr. ges. Physiol. Path. Stoffw., 6, 630-2.—When Ca(OH), was used in place of NaOH in Trommer's sugar test there was found to be a digression from the usual reduction process. To 5 cc. of urin in a test tube there are added 20 drops of Ca(OH), soln. (suspension of 40 g. CaO in 200 cc. water, and allowed to stand for 24 hrs.) and 5 drops of 10% CuSO<sub>4</sub> soln. The mixture is then boiled without shaking over a free flame until the froth reaches the middle of the tube. Every urin causes some discoloration, usually yellow-gray or blue-gray. If sugar is present, the soln, becomes darker, then red or violet. The Cu suboxide first formed is next reduced to metallic Cu. If there is much sugar this metallic Cu may be formed during the b. 0.1% glucose and lactose gave the same result. Uric acid, urates, and urea gave negative results. Albumin did not interfere. The change to metallic Cu was not caused by glucose, since an aq. soln. did not produce it. Diabetic urins, containing much glucose and small amts. of other solids, did not react as well. Creatinine might be the cause of the 2nd stage of the reduction, which did not occur if an excess of glucose was present, but dilution with water or normal urin remedied this condition. The test was more sensitive than Trommer's test and about equal to Fehling's. The latter can yield negative results when the sugar content is very high. Nylander's test is more delicate, but often positive when glucose is absent. The quantity of sugar can be detd. approx. by dilg. fractionally with sugar-free urin until the test fails. The content of sugar obtained in this way agreed with the results by Robert's method. Ba(OH), also gave similar results. Using 5 cc. of urin, 0.5 g. of Ba(OH), and CuSO, the mixt. gave a Cu mirror and a deep blue soln. when heated. This was formed when 0.025 % of glucose was present. The red substance accompanying the metallic Cu has not been examined but seems to be due to a mother substance present in all urins. This is changed into another substance when boiled with Ca(OH), and CuSO4, which is changed further by glucose into the red compd. C. A. BRAUTLECHT.

The Application of the Autenrieth-Tesdorpf Method of Sugar Determination in the Urin to the Quantitative Determination of Sugar in the Blood. FORSCHBACH AND SEVERIN. Med. Klin. Univ., Breslau. Zentr. ges. Physiol. Path. Stoffw., 6, 665-74.—Fehling's soln. is unsatisfactory for colorimetric detns. of sugar in the blood (cf. C. A., 5, 2392). The Autenrieth-Tesdorpf method, based upon Bang's Cu soln., is as follows; 5-6 cc. of defibrinated human blood (defibrinated by a splinter of wood) were pipetted into a small glass-stoppered Erlenmeyer flask and weighed. Protein was removed by adding 35-40 cc. colloidal Fe(OH)<sub>3</sub> and Na<sub>2</sub>SO<sub>4</sub> soln. to bring up the vol. to 50 cc. 10-30 cc. of the clear colorless soln. (obtained by filtering under nor-

mal or reduced pressure or centrifuging) are made up to a vol. of 35 cc. by adding water. 5 g. K<sub>2</sub>CO<sub>3</sub>, 4 g. KNCS and 10 cc. Bang's soln. were then added and the whole boiled 3 min. in an Erlenmeyer flask, cooled, transferred quantitatively into a 50 cc. graduated glass and brought to the mark with 10% K<sub>2</sub>CO<sub>3</sub>-KNCS soln. This soln. was then compared colorimetrically with a test soln. in a Plesch chromophotometer. Variation of 3-5 min. in time of b. had no effect. The av. max. error in 3 duplicated detns. of blood containing a normal amt. of sugar (2 mg.) was 4.1%. With 3 mg. of sugar the av. max. error was 0.2%. Sugar added to the blood in quantities equiv. to low, normal and high amts. wasre covered quantitatively. The time required was 20-30 min.

The Estimation of Quinine as the Acid Citrate in Certain Organic Liquids. T. COCKBURN AND J. W. BLACK. Analyst, 36, 396–8.—A modification of Nishi's method. 250 cc. urin are treated with a large excess NaOH, warmed, extracted 3 times with Et<sub>2</sub>O, the latter evapd., dissolved in 2 cc. anhyd. Et<sub>2</sub>O and filtered into a tared flask, with which had been weighed a filtering tube. A satd. soln. of citric acid in anhydrous Et<sub>2</sub>O is added and after standing 24 hrs. the pptd. quinine acid citrate is filtered and weighed.

H. C. FULLER.

A Method for Studying the Chemistry of Cells. Martin Hahn. München. Münch. med. Wockschr., 58, 1385-8.—The method has been applied to the production of lactose by mammary glands. It is based on the removal of excess sugar by dialysis, thus preventing an inhibition of action of the lactose-forming ferment by an accountulation of lactose. Expressed fluid from cow's udder supplied with an antiseptic was allowed to stand in an incubator. Samples surrounded by parchment membrane sepg. them from a chamber of water containing an antiseptic showed considerable variation in reducing substance present for different periods of digestion. Samples unprovided with such membrane showed practically constant reducing power. The same was found true of aq. solns. of the acetone ppt. from the ext. Exts. of pancreas to which sugar was added showed const. reducing power without and greatly varying reducing power with the membrane.

G. R. Henry.

A Reaction for Anhydrides in the Protein Molecule. Bruno Bardach. Vienna. Chem. Zig., 35, 934.—Since CHI<sub>2</sub> in the presence of anhydride assumes a particular cryst. structure, it can be used for the detection of aromatic anhydrides in soln. or bound in the mol. The soln. to be tested must have a neutral reaction. To this is added dropwise a 2% alc. soln. CHI<sub>2</sub>. If this crystallizes in needles it indicates the presence of anhydrides particularly those of the type of euxanthone. With mol. anhydrides the above test would be negative (hexagonal crystals). The substance is then iodated with Lugol's soln. and NH<sub>2</sub> added and filtered after standing 1 hr. The filtrate is tested with CHI<sub>2</sub> and a positive result would indicate anhydrides in the mol. such as coumarin or polyhydroxyketones. The test can be applied to proteins and with Millon's reagent permits differentiating between phenolic hydroxides and hydroxy acids.

G. M. Meyer.

Red Coloration Given by Esbach's Reagent [with Urin]. C. GAZZATTI AND C. SARTI. Ist. farmacol. Modena. Arch. farmacol., 9, 319-29; through Zentr. Biochem. Biophys., 11, 887.—A pathological urin was found to give a red reaction with Rsbach's reagent, due to the presence of minimal amts. of (NH<sub>4</sub>)<sub>2</sub>S and NH<sub>3</sub>. When picric acid alone, instead of mixed with citric acid as above, was used, an orange-red color resulted. The color depends upon the reduction of picric acid to picraminic acid by the sulfide and is considered as the most sensitive test for that substance. Obviously glucose must be absent in such a test.

I. K. Phelles.

Reactions for Indole (VISSER). 17.

#### BOI ANY

Formation of Hydrocyanic Acid in the Germination of Seeds. C. RAVENNA AND M. ZAMORANI. Lab. chim. agrar. r. univ. Bologna. Gazz. chim. ital., 41, II, 74-81; cf. C. A., 5, 1633. Chas. A. Rouiller.

How Does the Cane Make the Sugar? JUAN J. CHAVANNE. Tucuman. Rev. ind. agr. Tucuman, 2, No. 1, 29-31.—Discussion of the present views of the photochem. action in the plant which produces the sugar.

G. A. ROUSH.

### PHYSIOLOGY.

Influence of Artificial Rise in Temperature upon Gastric Secretion in a Dog having a Pavlov Accessory Stomach. A. FISCHER. Univ. Berlin. Intern. Beitr. Path. Therap., 3, 86–100.—Secretion was diminished in some cases as much as 50% although the acid and pepsin concs. were not essentially changed. This decrease in secretion was considered to be due to loss in water.

C. A. BRAUTLECHT.

Transference between Living, Dead and Putrefied Tissues and the Fluids in which they are Immersed. Luigi Tomellini. Univ Genova. Arch. farm. sper., 12, 217-29.—Cond. measurements were made by the method of Benedicenti and Sabbatini, using living, dead and putrefied fragments of various organs. When the tissue is immersed in distilled H<sub>2</sub>O, the cond. of the latter increases, the increase depending upon the time of immersion. The longer the tissue has been dead, the more rapid is the transference of electrolytes to the surrounding medium, although putrefaction does not influence the max. cond. observed when an equil. is established between the osmotic pressure of the tissue and that of the surrounding medium. This fact may find application in medico-legal cases where it is desired to det. the time that has elapsed aince death occurred.

A. W. Dox.

Studies on Serum Proteins. BREINL. Pharm. Inst., deutsch. Univ. Prague. Arch. exp. Path. Pharm., 65, 309-14.—In agreement with Cervello (Ibid., 62, 357) the author found an increase in the globulin fraction after administration of antipyrin. This was found to be accompanied by a decrease in the albumin fraction. Extra corpus serum to which antipyrin had been added showed after 24 hrs. at body temp. an increase in the (NH<sub>4</sub>)<sub>x</sub>SO<sub>4</sub> fraction.

Walter A. Jacobs.

Researches on the Modifications of the Blood as a Result of the Extirpation of Thyroparathyroid Glands. P. Albertoni. Univ. Bologne. Arch. intern. physiol., 11, 29-37.—After complete thyroidectomy, the total vol. of blood of dogs was diminished to nearly the same extent as that of a dog which had fasted for the same length of time. The sp. gr. of the defibrinated blood was comparable to the sp. gr. of the blood of a starving dog with a slight increase above normal. The fibrin content of the blood was increased to 2-3 times the normal. Total blood protein was slightly increased. The NH<sub>3</sub> content showed variations within the normal limits. The glucose content (0.70%) was slightly below normal.

The Composition of the Normal Cerebrospinal Fluid. The True Nature of this Humor. W. MESTREZAT. Bull. soc. chim., 9, 683-8.—A mixt. of 20 normal spinal fluids, preserved by cold, contained in 1000 parts: H<sub>2</sub>O 996.7, solids 10.9, organic matter 2.1, ash 8.8, albumin 0.25, chlorides (as NaCl) 7.33, Na<sub>2</sub>O 4.35, K<sub>2</sub>O 0.25, CaO 0.1, MgO 0.05, Fe<sub>2</sub>O<sub>2</sub> 0.002, P<sub>2</sub>O<sub>3</sub> 0.03, SO<sub>2</sub> 0.03. Many detns., on 60 fluids obtained by puncture agree closely with these figures. The cerebrospinal fluid is not a secretion, but the result of a selective filtration from the plasma on the part of the choroid plexus. The principle constituents of the blood serum, albumin, fibrinogen, fats, lecithin (all colloids), are not found at all or only in traces, while Cl is more abun-

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dant in the spinal fluid than in the serum. It differs from the transudates which contain albumin, etc., in large amt. and resembles the humors of the eye and inner ear.

J. F. LYMAN.

The Position Occupied by the Production of Heat in the Chain of Processes Constituting a Muscular Contraction. A. V. Hill. J. Physiol., 42, 1-43.—When a muscle is stimulated some substance is liberated which acts on the colloidal tissues of the muscle causing a tension proportional to its conc. The substance is then destroyed or returned to its original position (oxidized), the heat evolved being proportional to the amt. of substance (for instance lactic acid) in question. Muscle then is not a heat engine.

J. F. Lyman.

The Pigments of the Blood. Reduction of Hematoporphyrin with Tin and Hydrochloric Acid. Hematopyrrolidic Acid. O. PILOTY. Mon. sci., [4] 24, 711-23.—Methods are described for the prep. of hemopyrrole, hemopyrrolecarboxylic acid, and hematopyrrolidic acid from hematoporphyrin. Hemopyrrole, CaH12N, in acid soln., is vigorously attacked by the O of the air and transformed into a pigmented comp. which Nencki has considered as isomeric with urobilin. Hemopyrrolecarboxylic acid, C<sub>2</sub>H<sub>12</sub>NO<sub>3</sub>, yields on oxidation a mixt. of all the pigments which are formed when the reduction products of hematoporphyrin are exposed to the air. When hemopyrrolecarboxylic acid in soln. in Et.O is treated with dry air a brown amorphous ppt., having the formula C18H2N2O4 forms. Its alc. soln. is affected by the air, green platelets sepg. These were not collected in sufficient amt. for analysis, but gave a spectrum similar to that of chlorophyll. At the end of several months an acid ethereal soln. of hemopyrrolecarboxylic acid kept in contact with the air deposits a deep red-brown substance, green in reflected light. This mass is composed of a mixture of very closely related pigments which are very difficult of crystn. By the aid of MeOH and Et.O a violet pigment insol. in MeOH, a green pigment easily sol. in MeOH but pptd. by Et<sub>2</sub>O, and finally a violet pigment (probably C<sub>12</sub>H<sub>22</sub>N<sub>2</sub>O<sub>2</sub>) sol. in MeOH and pptd. by the addition of much Et<sub>2</sub>O were sepd. Two compds. are produced by the action of HNO<sub>2</sub> on hemopyrrolecarboxylic acid: (1) A colorless compd., C<sub>2</sub>H<sub>10</sub>N<sub>2</sub>O<sub>4</sub>; (2) a compd., CaHaNO4, probably identical with Kuster's hematinic acid. Desoxyhematoporphyrin, Cathan N.O., is the 1st product of the reduction of hematoporphyrin in HCl. A blue pigment, apparently isomeric with desoxyhematoporphyrin, was obtained by reducing hematoporphyrin in AcOH with Zn. Desoxyhematoporphyrin dissolved in conc. HCl and reduced in the cold with Zn powder, yields principally 3 products: (a) Hemopyrrole, (b) hemopyrrolecarboxylic acid, (c) hematopyrrolidic acid (probably C<sub>1</sub>, H<sub>22</sub>N<sub>2</sub>O<sub>2</sub>). Theoretically 100 g. hematoporphyrin yields on reduction 20 g. of (a), 26 g. (b) and 42 g. (c). The author obtained from 100 g. hematoporphyrin, 10, 20 and 34 g. of these derivs., resp. (73% of theory). J. F. LYMAN.

The Allantoin-Purine Excretion of the Monkey. A. HUNTER AND M. H. GIVENS. Proc. Am. Physiol. Soc., 1910, 15-6; Am. J. Physiol., 27.—Allantoin was isolated from the urin of a monkey by the method of Wiechowski. From 500 cc. monkey urin no uric acid could be isolated and only 4.5 mg. of N in the form of purine bases.

J. F. LYMAN.

Physiological Chemistry. Ludwig Pincussohn. Fortschritte Chem., 4, 195-209.—Review for the period Dec., 1910-May, 1911, under the headings: phys.-chem.; chem.; metabolism; ferments.

J. J.

Gum and Gelatin (TIEBACK). 28.

Lipase in the Fat of the Fowl (PENNINGTON, HEPBURN). 12.

New Leucomaine (CORREAL.) 10.

## PATHOLOGY

Influence of Hydrocyanic Acid on the Elimination of Urinary Sulfur. R. MAGNANIMI. Univ. Sassari. Arch. farm. sper., 12, 210-6.—The urin of animals poisoned by HCN contains no albumin nor sugar. Total N and S are decreased, but the latter shows no disturbance in the relative proportions of acid and neutral S until the following day when the acid S increases and the neutral S decreases slightly. This fact is of importance, since it is known that the neutral S increases in dyspnea, lack of O and other respiratory disturbances, as well as under the influence of the so-called protoplasmic poisons, narcotics containing Cl, and probably other conditions where the non-urea N is increased. If neutral S is regarded as an intermediary product in the formation of sulfates, the author's results are somewhat difficult of interpretation. They are, however, in accord with Benedikt's hypothesis that neutral S, unlike H<sub>2</sub>SO<sub>4</sub>, is comparable to uric acid in that it is independent of protein metabolism.

A. W. Dox.

Meaning of Urochromogen for Prognosis and Therapy of Pulmonary Tuberculosis.

Moriz Weisz. Vienna. Manch. med. Wockschr., 58, 1348-52.—W. reports results of 6 yrs. work with the KMnO<sub>4</sub> test for urochromogen in urin of patients suffering with pulmonary tuberculosis. It is first necessary to exclude other causes for the appearance of urochromogen. These are (1) other toxemias such as typhoid fever, measles, scarlet fever, septicemia, etc., and surgical tuberculosis; (2) narcosis; and (3) injection of tuberculin. The reaction first becomes positive in pulmonary tuberculosis when there are other signs of toxemia. Excretion of urochromogen is due to insufficient oxidation and it runs parallel to the neutral S of urin. It may indicate a low resistance to the toxin of the tubercle bacillus or a spreading lesion with increased production of toxin. It indicates an unfavorable prognosis. W. advizes that the test be used (1) as a help in prognosis, (2) to det. which cases shall receive tuberculin treatment, and (3) to serve as a check on the value of the therapeutic measures adopted.

G. R. HENRY.

Colloidal-chemical Analysis of Rephritis. M. H. FISCHER. Z. Chem. Ind. Kolloids, 8, 201-8 (1911); through Chem. Zentr., 1911, I, 1873; cf. C. A., 5, 2393.—A continuation of work published in the author's book on edema. The acute nephritic kidney has the characteristic of cloudy swelling and may thus be explained on the basis of colloidal chemistry. All conditions which evoke cloudy swelling (acid production in the cell) cause the kidney to break down into epithelial, granular and hyalin cylinders. Increasing the acid conc. will cause the 1st of these cells to change to the 2nd, then 3rd. Hyalin cylinders can be changed back again by adding a salt to the acid soln. The acid in the kidney cell causes the solubility of the albumin, and its appearance in the urin. There follows a discussion of the foundation for this assumption and finally some general observations on the different clinical forms of nephritis and the nature of diabetes.

Further Studies on the Experimental Production of Goiter. The Etiology of Goiter as a Colloid-chemical Problem. Eugen Bircher. Aarau. Z. exp. Path., 9, 1-38; through Chem. Zentr., 1911, II, 46.—Led by the conviction that goiter is produced by a colloidal toxin contained in certain drinking waters (cf. Deut. Z. Chir., 103) and supported by expts. on rats and monkeys, B. reached the following conclusions: Goiter toxin is an hydrosol of organic nature distributed in well water. The attempts to dialyze it showed its colloidal nature. The dialysate was inactive, the undialyzed portion producing goiter. The hydrosol is but little opalescent or fluorescent. On conc. of its solns. it is pptd. along with the Ca salts which sep. Filtration expts. indicated it to be composed of a suspension and an emulsion phase. The first is held by a Berke-

land filter and produces the symptoms of cretinism. In rats it disturbs growth and after 12-15 months ingestion it produces small goiters. The emulsion phase passes through the filter and causes, after 3 months, decided symptoms. If rock from an inactive stratum is placed in contact with water from a goiter well for 48 hrs. the toxin is absorbed by the rock so that rats given this water do not develop goiter. After several months the rock becomes satd, with the toxin and no longer absorbs it. Ordinary water was not rendered active by two weeks contact with rock containing the toxin.

Walter A. Jacobs.

A Few Physico-chemical Properties of Human Milk. Jeannette Polenaar and H. Pilippo. Pharm. ther. Lab.; Univ. Leyden. Z. exp. Path., 9, 138-45; through Chem. Zentr., 1911, II, 39.—Detns. of viscosity, elec. cond. and f. p. were made during the first 20 weeks on the milk of nursing women. On the av. no differences were observed in the viscosity between the milk of normal and abnormal cases (scanty secretion, albuminuria and acute and chronic diseases). In almost all cases during the first day after labor a pretty regular decrease in viscosity occurred. The cond. was in abnormal cases almost always increased, least so in cases of albuminuria and most in cases of scanty milk supply. In normal cases, particularly during the first week, a regular decrease in the cond. was observed.

Walter A. Jacobs.

The Viscosity of the Blood in Healthy and Sick Infants. F. Lust. Univ. Kinderklinik., Heidelberg. Arch. Kinderheilh., 54, 260-78.—Viscosity detns. were made with a Hess viscosimeter. The av. value for the blood of a healthy infant was found to be 3.8. During the postnatal period the viscosity increased very considerably; there was also an increase in the form elements, hemoglobin and generally of the entire dry substance of the blood. In the  $\frac{1}{2}$ - to 11-year period the viscosity approaches that of adults (mean value 4.1). Anemic infants show regularly a lower viscosity. No constant ratio between viscosity and hemoglobin value was found. All conditions leading to marked cyanosis increase the viscosity due to the accumulation of CO<sub>2</sub>. Very close relationship was observed between viscosity and the water content of the blood, but not of such a nature as to permit exact deductions from one to the other. Influences which temporarily affect the water content of the blood such as changes in nutrition, particularly the ingestion of food rich in carbohydrate, show themselves in a corresponding decrease in the viscosity. Intravenous or subcutaneous injection of physiol. NaCl soln. more strongly affects the viscosity. Chronic nutritive disturbances show little change in the viscosity. Acutely occurring disturbances, however, show decided variations, and where large water losses occur may increase it more than 100%. WALTER A. JACOBS.

The Action of Asphyxia on the Spinal Animal. G. C. Mathison. J. Physiol., 4x, 416-49.—The similarity between the effects produced by the administration of N and by asphyxia shows that lack of O is the main factor in asphyxia in the spinal animal. Lack of O, excess of CO, and weak acids injected into the blood are all capable of stimulating the spinal vaso-motor centers.

J. F. LYMAN.

Analysis of a Biliary Calculus. H. Montlaur. Bull. sci. pharmacolog., 18, 19-20.—The calculus weighed 0.83 g. and consisted mainly of Ca oxalate with traces of H<sub>2</sub>PO<sub>4</sub>, Mg, K, a small quantity of fat and of an undetd. substance insol. in acids but destroyed by heat.

V. K. Chesnut.

Carbophosphates of the Saliva. Their Metabolism in the Formation of Dental Tartar. M. Barille. Schweiz Wochschr., 49, 362.—It has been shown that carbophosphates of the alkaline earths are transformed in the milk, urin, urinary calculi, etc., and as these substances exist in the saliva they are probably transformed by

biological mechanism and deposited on the teeth as tartar. Dentifrices containing soap should be avoided, preference being given to those of acid nature.

H. C. FULLER.

### PHARMACOLOGY.

Viscosimetric and Cryoscopic Variations in the Blood after Administration of Antipyrine, Phenacetin and Antifebrin. Guido M. Piccinini. Univ. Bologna. Arch. farm. sper., 12, 193-209.—The cryoscopic point of blood serum rises after the introduction of antipyrine, phenacetin or antifebrin, then descends to the original figure. The variations in viscosity follow the same curve. In the case of antipyrine a decided difference is noted between the results obtained with serum and with defibrinated blood, and the author points out the importance of including observations on the latter when reporting analyses.

A. W. Dox.

Pharmacological Properties of Adalin (Bromodiethylacetylurea). EDUARDO FILIPPI. Ist. Stud. Sup. Firenze. Arch. farm. sper., 12, 233-40.—Adalin is a sedative
capable of inducing a light but enduring sleep. It exerts no action on the heart, and
its toxicity is very slight. The substance is eliminated in part unchanged; in part as
a brominated acid of the probable formula (Et)<sub>2</sub>CBrCOOH, 87-89°. Only after administration of toxic doses is there any cleavage of the brominated fatty acid and a
formation of alk. bromide.

A. W. Dox.

The Relation between the Chemical Structure and the Physiological Action of Organic Preparations. P. J. Montagne. Leiden. Pharm, Weekbl., 48, 857-74.——Interesting and instructive lecture with well chosen illustrations.

V. E. HENDERSON.

Opium-Urethan Combination. Chassia Rappoport. Med. chem. Pharm. Inst., Univ. Bern. Z. exp. Path., 9, 39-48; through Chem. Zentr., 1911, II, 40.—From the observations of Lindemann (Ibid., 7, 725; also Burgi, Ibid., 8, 523) that the combination morphine-urethan leads to a mutual intensifying of their actions it may be concluded that the combination opium-urethan not only results in an addition but to a mutual intensifying of their sep. actions. For the rabbit 2.5 g. tinctura opii  $(N_0)$  was found to be the minimal anesthetizing dose. In agreement with the above assumption the following combinations were found to produce anesthesia. 0.5  $N_0$  + 0.04  $N_u$  ( $N_u$  = minimal anesthetizing urethan dose), 0.5  $N_u$  + 0.04  $N_0$  and 0.2 $N_0$  + 0.2  $N_u$ . Walter A. Jacobs.

The Action of Phosgene on the Human and Animal Organism. ROBERT MULLER. Elberfeld. Z. exp. Path., 9, 103-25; through Chem. Zentr., 1911, II, 41.—For the study of phosgene intoxication clinical experiences with humans and the results of poisoning by decomposed CHCl<sub>s</sub>-narcotic mixts. were discussed. The facts here developed were confirmed by toxicity detns. on rats. Inhalation expts. were performed in a Dresser app. (Arch. exp. Path. Pharm., 37, 375) with a volatilized COCl<sub>2</sub> soln. in C<sub>2</sub>H<sub>2</sub>. Control expts. showed that the toxic effects of the quantity of CaHa used could be sharply differentiated from those of the COCl, and also were less pronounced than those of the latter. The toxic action of COCl, is less dependent upon the amt. of COCl, which enters the organism than upon its conc. and the duration of its action. With a conc. of 0.05o. 2 vol. % COCl, in the air the animals succumb with severe lesions in a few hrs. With a conc. of 0.0123 vol. % they tolerate repeated inhalations and survive for a considerable time. The histo-pathological conditions are similar to those described by others for the toxic after effects of CHCl2. By per os administration the action is of an intense local caustic nature. The partition coeff. for COCl, between C.H. and water was found to be 0.764: 0.736. WALTER A. JACOBS.

Fibrinogen of the Blood as Influenced by the Liver Necrosis of Chloroform Poisoning. G. H. Whipple and S. H. Hurwitz. Hunterian Lab. of Exp. Path., Johns Hopkins Med. School, Baltimore. J. Exp. Med., 13, 136-61.—CHCl<sub>2</sub> anesthesia for 2 hrs. or more causes more or less central liver necrosis in dogs. The fibrinogen of such an animal estimated at intervals is found to show a decrease corresponding to the amt. of liver necrosis. By administering CHCl<sub>2</sub> the fibrinogen may be almost eliminated from the circulating blood, thus rendering it practically non-coagulable. The liver can recover and return to the normal condition in about 10 days, this being accompanied by the reappearance of fibrinogen which after recovery may be above normal. In several cases of CHCl<sub>2</sub> poisoning the Ca of the blood was normal or slightly increased and the thrombin normal. Fibrinogen is either formed in the liver or is wholly dependent upon hepatic activity for its production.

Walter A. Jacobs.

The Effect of Potassium Salts on the Circulation with Special Reference to the Production of Heart Block. G. C. Mathison. Proc. Physiol. Soc., 1910, 19-20; J. Physiol., 41.—When both brain and spinal cord are destroyed, solns. of KCl injected into the carotid produce a rise of blood pressure mainly due to vaso constriction. When injected into the jugular vein the salts act first on the heart causing diastolic arrest and a fall of pressure. With the proper dosage heart block may be produced.

J. F. Lyman.

The Effects of Extracts of Different Parts of the Pituitary Body. J. L. MILLER, D. D. LEWIS AND S. A. MATHEWS. Proc. Am. Physiol. Soc., 1910, 17-8; Am. J. Physiol., 27.

J. F. Lyman.

Normal and Pathological Physiology of the Chromoffin Tissue of the Adrenals. Gustav Bayer. Innsbruck. *Ergebnisse allg. Pathol.*, 14 (II), 1-132.—A general review, covering particularly the chemistry and physiology of epinephrine.

H. G. WELLS.

Hematogenous Stimulation of Gastric Secretion by Hydrochloric Acid Extracts of Large Glandular Organs of the Body and Intestinal Contents. O. Emsmann. Univ. Berlin. Intern. Beitr. Path. Therap., 3, 117-30.—The HCl exts. of the pylorous, duodenum, jejunum, mucous membrane of the ileum, liver, pancreas and contents of large intestin stimulated gastric secretion constantly and to a great degree. The exts. of mucous membrane of the large intestin, spleen, submaxillary, parotid and mucous membrane of small intestin caused less stimulation, while kidney ext. caused none. Subcutaneous injection gives max. action; per os only a min.

C. A. B.

## 11b. IMMUNOCHEMISTRY.

#### H. GIDEON WELLS.

Venoms and Antivenoms. MAURICE ARTHUS AND BOLESLAWA STAWSKA. Compt. rend., 153, 355-7.—The neutralization of venoms by antivenoms was instantaneous and thus was more like a chem. reaction than a ferment action. A. and S. used in these tests cobra venom, the venom of Lachesis lanceolatus and Crotalus terrificus, neutralizing them with their corresponding antivenoms and injecting into the ears of rabbits. The antivenoms of the last 2 venoms mentioned above, when mixed with their corresponding venoms, prevented coagulation of fibrinogen solns. (peptone blood of dog, oxalate and fluoride plasmas of horse and NaCl solns. of fibrinogen) almost instantly after mixing.

H. J. CORPER.

The Specificity of Antivenom Serums. Anticobra Serum and Hanadryas and Krait Venoms. MAURICE ARTHUS. Compt. rend., 153, 394-7.—In the action of the 3 venoms, cobra (Naja tripudious), hamadryas (Naja bungarus), and krait (Bungarus)

cosrulous) upon rabbits the author noted a certain amt. of similarity which led him to test the antitoxic value of cobra antivenom serum toward the hemadryas and krait venoms. He found that it takes just about 15 times as much cobra antivenom serum to neutralize the last 2 venoms as to neutralize cobra venom.

H. J. CORPER.

The Effects on the Protoplasm of Nitella of Various Chemical Substances and of Microbrganisms Introduced into the Cavity of the Living Cell. M. A. BARBER. Lab. of Path. Bacteriology, Univ. of Kansas. J. Inject. Dis., 9, 117-29.—The author injected into the protoplasm of Nitella various chem. substances with the following results: Strychnine, alc., As, methylene blue, metallic Hg and quinine had very little effect upon the protoplasm; small doses of satd. CuSO<sub>4</sub> introduced into the vacuole of the cell produced little effect; CHCl<sub>2</sub> and ether exerted a powerful local effect and even a very small amount produced death of considerable protoplasm, but they had not so great a general effect as HgCl<sub>2</sub> or osmic acid. CH<sub>2</sub>O was also powerful in its action. The interior of certain living plant cells (Nitella, Vaucheria, Saprolegina, Achlya and Dictypchus) is a good medium for the growth of bacteria, yeasts and sporotrichum, there apparently being no antibacterial property in their protoplasm and the death of the cell being due to parasitism rather than to toxins. H. J. C.

Studies on the Chemistry of Anaphylaxis. III. Experiments with Isolated Proteins, Especially those of the Hen's Egg. H. GIDEON WELLS. Path. Lab., Univ. Chicago. J. Inject. Dis., 9, 147-71.—By means of the anaphylaxis reaction Wells demonstrated that ovovitellin from the yolk of hen eggs was distinct from cryst. egg albumin, and ovomucoid from hen egg white and also from vitellin from turtle eggs. Ovomucoid, in spite of b. and pptn. with alc., produced anaphylaxis, and was distinct from cryst. egg albumin, repurified "globulin" of egg white, and ovovitellin, all from hen eggs. The "globulin" fraction had about the same degree of intoxicating and sensitizing power as cryst. egg albumin and the reaction against each other and against themselves was about equal; however, they have specific and distinct antigens besides the common antigen, not separable by (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, as shown by the fact that guinea pigs sensitized with several antigens can be satd. with one of these antigens and then will react severely to one of the others. Thus the author found 5 antigens in hen's eggs, 4 in the white and 1 in the yolk. Crude egg white had less sensitizing and intoxicating powers than corresponds to the activity of the isolated proteins; the nature of the inhibiting substance was not found. The refractory condition of guinea pigs to sensitization by means of a protein after feeding this protein, seems to be reached more easily with vegetable proteins of the natural food (corn, oats) than with animal proteins. Caseinogen gives a strong anaphylaxis reaction; the casein of cow milk and of goat milk react against each other. Na nucleinate from cod sperm does not give anaphylaxis reactions while the albumin does. Histone from the same source produces no anaphylaxis reaction, is toxic to guinea pigs and 56° does not destroy the toxicity. Mucin from the pig's stomach produces typical anaphylaxis and is specific against pig's blood serum or extracts of pig muscle. Two years autolyais of human placenta extract did not destroy its antigen or specificity. Three years' digestion of bovine serum with trypsin did not quite destroy its sensitizing power.

H. J. CORPER.

The Bactericidal Action of Quinone and other Phenol Oxidation Products as Determined by the Rideal-Walker Method. WILLIAM THALHEIMER AND BARTON PALMER. Univ. Virginia. J. Inject. Dis., 9, 172-80.—In expts. with various samples of phenol the authors found that some highly colored preps. had a more powerful bactericidal action than colorless phenol, using the Rideal-Walker method of standardization.

This led them to test various pure phenol oxidation products. The Rideal-Walker phenol coeff. of quinone is 160; that of hydroquinone is only 2.5. The activity of quinone is also reduced when joined with the less active phenol to form phenoquinone (coeff. 44).

H. J. CORPER.

A Comparison of the Bactericidal Action of Quinone with that of some of the Commoner Disinfectants. WILLIAM THALHEIMER AND BARTON PALMER. Univ. Virginia. J. Inject. Dis., 9, 181-9.—In order to compare the bactericidal action of the commonly used disinfectants with quinone the authors made use of previous figures by others and made the following observations, using the Rideal-Walker method (preceding abst.). I and alc. (in strengths above 50%) are efficient bactericidal agents; KCN and HCN have a weak bactericidal action. The phenol coeff. of crude phenol is 4, tricresol is 2.2, and of paracresol is 2.8, thus making it impossible that a combination of all 3 cresols is more powerful than any one of them.  $\beta$ -Naphthol and NaOH neutralize one another's bactericidal action, thus the inconsistency in the use of the former as an intestinal antiseptic.

Application of Physical Chemistry to the Doctrin of Immunity. Antigens and Antibodies. S. Arrhenius. Chem. News, 104, 55-8.

J. J. M.

Eclampsia as an Anaphylactic Phenomenon. FELLANDER. Bakt. Lab. med. Staatsanstalt, Stockholm. Z. Geburtshilfe Gym., 68; through Z. Immunität., Ref., 4, 366.—The attempt to produce anaphylaxis with placental exts., milk, or fetal serum from the same species, was always unsuccessful. Neither did injection of guinea pig placenta make guinea pigs sensitive to guinea pig serum, or reversely. The symptoms of eclampsia are entirely dissimilar to those of anaphylaxis.

H. G. W.

The Anaphylactic Theory of Toxemia of Pregnancy. JOHNSTONE. Dept. Midwifery, Univ. Edinburgh. J. Obst. Gyn. British Empire, 19; through Z. Immunität., Ref., 4, 366.—Attempts to produce anaphylaxis with exts. of the homologous placenta gave negative results.

H. G. W.

Protozoan Toxins (Sarcosporidiotoxin). TEICHMANN AND BRAUN. Arch. f. Protistenk., 22, 351.—Sarcosporidia, which occur especially frequently in the muscles of deglutition of sheep and goats, contain a strong toxin. Injected into rabbits subcutaneously, an ext. of these parasites causes death in a few hrs., but it is not poisonous when taken into the stomach. The toxicity is reduced by heating at 60°. Immunity is readily obtained in rabbits, and their serum is antitoxic. Other animals are not susceptible to this toxin, but their blood contains no antitoxin. H. G. W.

Relation of Complement of Serum to Surface Tension of Serum. Secale. Inst. Pathol. Gen. Univ. Genoa. Patol., 1, 709–10; through Z. Immunitat., Ref., 4, 385.—Traube's statement that heating of serum to 55°, or aging until the complement has disappeared, causes an alteration in the surface tension is confirmed. Such inactivated serum shows by ultramicroscopic exam. aggregates of particles with active motion. Removal of these by centrifugation restores the surface tension almost to its original value. Addition of the sedimented micellae to fresh serum causes a lowering of surface tension, but the complement action is not affected thereby. Therefore it is concluded that the substances formed in old or heated serum which lower the surface tension have nothing to do with the complement function.

H. G. W.

Anaphylaxis against Proteins of Homologous Species. HERTLE AND PREIFFEE. Inst. Gericht. Med., Univ. Graz. Z. Immunität., 10, 541-9.—The authors sought to learn if anaphylaxis could be produced with exts. of tissues of homologous animals, using cell suspensions of guinea pig organs washed free from blood proteins by centrifugation with 0.125 N NaCl soln. Sensitization of guinea pigs with emulsion of kidney

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cells from guinea pigs renders them reactive to subsequent doses of the same material. Less marked results were obtained by sensitizing with guinea pig adrenal and testicle emulsions, and chiefly after repeated sensitizing injections, followed by an interval of 40 days before the intoxicating dose of guinea pig kidney emulsion; the homologous emulsions (adrenal, testicle) did not cause symptoms in these animals. Crushing or slicing of one kidney, after ligation of its vessels, rendered guinea pigs sensitive to subsequent injection of emulsion of guinea pig kidney.

H. G. W.

Hypersensitivity and other Toxicoses from Acute Parenteral Destruction of Proteins. H. PFEIFFER. Inst. Gericht. Med., Univ. Graz. Z. Immunität., 10, 550-710.—The urin of guinea pigs contains traces of toxic substances, which are greatly increased during anaphylactic shock and in proportion to the severity of the reaction, not appearing if some antigen different from the sensitizing antigen is introduced, or during antianaphylaxis expts. Peptone poisoning causes the same substances to appear in the urin. Injection into guinea pigs of these urinary poisons causes the same local and general symptoms as are characteristic of the reactions of hypersensitiveness, and survival from intoxication with the urin removes or reduces any existing hypersusceptibility. Animals in the antianaphylactic condition are also refractory to the urin poisons. Uremia produced by nephrectomy results in the same symptoms and anatomical changes as anaphylaxis. The toxicity of the urin in anaphylaxis is not dependent on any of the usual constituents, but apparently there are 2 distinct poisons present (with local and systemic effects respectively) which are probably abiuret compds. derived from proteins produced in the proteolysis of anaphylactic shock, and therefore identical with or closely related to the anaphylatoxin. As similar substances are normally present in urin in small amts., anaphylaxis may be looked upon as an "over-production uremia." Hemolytic foreign sera also produce symptoms and anatomical findings similar to anaphylaxis, and the urinary toxicity is likewise increased. Poisoning with such toxic urin makes animals refractory to the hemolytic poison. Hence it is concluded that hemolytic sera are not directly toxic, but only through disintegration by a complement-amboceptor proteolysis. Photodynamic action of light after injection of fluorescent substances is brought into the same category for the same reasons, and is interpreted as a toxicosis resulting from parenteral disintegration of proteins through photodynamic action. Death from extensive burns is likewise a toxicosis from protein destruction.

Remarks on the Above Article of H. Pfeisser. A. Biedl and R. Kraus. Z. Immunität., 10, 711-4.—Controversial. H. G. W.

## 12. FOODS.

W. D. BIGELOW.

The Relation of Food Chemistry to Public Health and Well-being. A. BEYTHIEN.

Z. Nahr. Genussm., 22, 8.—An address.

A. F. SEEKER.

Our Work of Enlightenment. Alfred Hasterlik. Z. Nahr. Genussm., 22, 73-81.—An address. P. B. Dunbar.

The Dutch Alimentary Codex. A. J.-J. VANDEVELDE. Rev. gén. chim., 14, 269-77. J. J. M.

Supervision of Commerce in Agricultural Products in Portugal. Z. öffent. Chem., 17, 313-4.—Sections of laws governing the production of wine, and detailed statements of what constitutes adulteration of same.

D. S. Pratt.

Berlin Experiment Station for Hygienic and Industrial Purposes. PROSKAUER. Chem. Ztg., 35, 899-900, 928-9.—Analyses are given of meat exts., beer, animal fats and meal, and street dust. "Automors" appeared to be made by heating conc.  $H_xSO_4$  with a tar oil containing phenol. It is similar to "sanatol." Traces of Zn were found on coconut cake that had caused death when used as cattle food. This was due to contamination with Zn ore on shipboard. "Antidustin" was found to be an aq. soln. of rosin, rosin oils, etc. (made sol. by treatment with alkali) and about 20% alc. A material for cleaning tin was crude NaOH soln. taken up by starch. D. S. Pratt.

Report of the Experiment Station at Nürnberg in 1910. H. Schlegel. Chem. Ztg., 35, 998, 1017. J. M.

The Final Report of the Royal Commission on Human and Animal Tuberculosis. J. WILBERFORCE GREEN. Brit. Food J., 13, 141.—Man is notably susceptible to bovine bacilli, and much of the tuberculosis of childhood is attributed to bovine bacilli in milk.

A. W. Broomell.

The Occurrence and Permanence of Lipase in the Fat of the Common Fowl (Gallus domesticus). M. E. Pennington and J. S. Hepburn. U. S. Dept. Agr., Bur. of Chem., Circ. 75, 1-7.—The acidity of the crude abdominal fat, rate at which various esters are hydrolyzed by an aq. ext. of the fat, the further hydrolysis of the esters after neutralization, during incubation, and the influence of chem. constitution on the rate of hydrolysis of the esters, indicate that lipase is present in the fat. During life the lipase exists in zymogenic form, and with fowls just killed there is little lipolytic action. As the storage time increases, the enzyme becomes active, and the acidity increases. These changes are more rapid the warmer the storage temp. Lipase was found in fowls stored at 0° for 89 mos.

The Oxidation of Chicken Fat by means of Hydrogen Peroxide. JOSEPH S. HEPBURN. U. S. Dept. Agr., Bur. of Chem., Circ. 75, 7-11.—Applying the  $H_2O_2$  method of Dakin (C. A., 4, 2635) the petroleum ether ext. of chicken fat was oxidized, and the fat consts. detd. before and after treatment. There is always an increase in the acidity, and the sapon. no. and Hehner no. usually increase simultaneously. These same changes occur naturally in chickens stored hard frozen, indicating that there is a formation of lower fatty acids and ketones from the satd. acids originally present.

H. S. BAILEY.

The Composition of True Roquefort Cheese. ARTHUR W. Dox. Storrs. Agric. Expt. Sta. Z. Nahr. Genussm., 22, 239-42.—Samples of Roquefort cheese from each of the 8 leading foreign manufactures were analyzed. These analyses (table given) show that the % of H<sub>2</sub>O, fat, protein, ash and NaCl is fairly const.; and, except in the case of NaCl and ash, similar to that found for Gorgonzola and Stilton cheeses.

H. S. BAILEY.

The Composition of Acid Curd (Kuhtopfen). G. Biro. Kgl. Chem. Versuchsstation, Magyaróvár. Z. Nahr. Genussm., 22, 170-1.—This product is obtained by pptg. the neutral casein of cow's milk with acids, generally the naturally formed lactic acid, in distinction to that obtained by using rennet. H<sub>2</sub>O, total acidity as lactic, casein, fat, ash, and n of the fat were detd. It is used in large amts. to mix with a similar product from sheep's milk in a soft cheese called "Liptauer Topfen." The ratio of CaO: N may be used to detect this addition. In the curd from sheep's milk this ratio is 0.242:0.320 while from cow's milk it is only 0.041:0.056. The ratio P<sub>2</sub>O<sub>6</sub>: N was detd. as 0.1148: 0.193.

Examination of Lactose and By-products of the Lactose Industry. IV. A. Burr AND F. M. BERBERICH. Chem. Ztg., 35, 803-4.—From 100 kg. of whey about 5 kg. of

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albumin are obtained. Because of this high %, whey mixed with potatoes, etc., low in N, is an especially good ration in fattening swine. More often it serves for the manuf. of cheese, "Zigerkäse," or as food either fresh or baked with meal. Analyses of this product, of the residue left in the filter-press used in clarifying, and of the mother liquor from which the milk sugar is obtained, are given in detail. D. S. Pratt.

The Action of Ozone on Milk and Dairy Products. W. FREUND. Chem. Ztg., 35, 905-6.—The effect of O<sub>2</sub> as a sterilizing agent for milk, cream, butter and soft cheese was studied. The rise in acidity of milk treated with O<sub>2</sub> is retarded, but chem. changes which cannot be corrected by aeration occur in the milk, rendering it unfit for the market. Treatment of milk with ozonized air containing 0.0027 g. O<sub>2</sub> per 1. for 2<sup>1</sup>/<sub>4</sub> min. produces a brown discoloration; after treatment for 55 min. a slight flocculence appears on the surface. These are evidences of chem. decomp. of a deep-seated character, consisting probably in a breaking up of fats and perhaps a further decomp. of the free fatty acids and albuminous substances, injurious results which are increased when the milk is delivered to the O<sub>2</sub> in the form of spray. Undesirable results are likewise obtained when cheese is treated with ozonized air. Butter made from cream which was so treated was churned in less time than butter from untreated cream, and was practically free from undesirable taste.

Chemistry of Milk and Whey Products in 1910. II. M. SIEGFELD. Chem. Ztg., 35, 986-7.

J. J. M.

Milk as a Powder. F. Marre. Rev. gén. chim., 14, 229-32.—A review of the various processes and patents for the evapn. and desiccation of milk. The method proposed by Fernando, Leconte and Lainville, in which the H<sub>2</sub>O is frozen instead of boiled out, is briefly described.

H. S. Balley.

The Detection of Peanut Oil in Olive Oil. S. Fachini and G. Dorta. Chem. Ztg., 34, 994.—The method differs from that of Tortelli and Ruggeri (Ibid., 34, 689) in that acetone is used as the medium for sepg. archidic acid from the sol. acids in place of petroleum ether or EtOH. Solid satd. fatty acids are difficultly sol., unsatd. acids are easily sol. and esterification cannot take place in acetone. Saponify 20 g. sample in alc. KOH, evap., dissolve the soap in H<sub>2</sub>O, liberate the acids with H<sub>2</sub>SO<sub>4</sub>, warm to melt the acids to form a clear layer on top of soln., collect on a moist filter, dissolve in 150 cc. warm acetone (b. 56-57°) without drying and add H<sub>2</sub>O drop by drop until turbidity appears. If the soln. should become permanently turbid or sep. into 2 layers, add acetone drop by drop until the soln. remains clear at 40-45°. Then allow to cryst. and cool slowly to 15°. After 1 hr. filter off the crystals and wash with 10 cc. dil. acetone (32 H<sub>2</sub>O + 68 acetone by vol.). Arachic or lignoceric acids are then detd. according to Tortelli and Ruggeri.

E. J. WITZEMANN.

Ripe and Unripe Bananas. R. REICH. Hyg. Inst., Leipzig. Z. Nahr. Genussm., 22, 208-26.—An elaborate investigation of various types of bananas and banana flours. Two kinds are used for flours, the plantain and the fruit banana. The former contains, both when ripe and unripe, a large % of starch and little sugar, while the latter when ripe has changed nearly all of the starch into sugar. The common form used as fruit belongs to the genus Musa paradisiaca of which a large number of varieties exist. The plantain is used in Surinam as food, but only the fruit banana, called "bacove," is cultivated. When dried ripe, "Great Michel" bananas contain about 1% sucrose, 60% invert sugar, and 3% starch. The same from green fruit contains about 2% invert sugar and 70% starch. The ash is high in K in both cases. The banana peels turn yellow or red as the sugar begins to form. Various characteristic esters are then present. The Pikien Missifinger bacoves are only about 3 inches long, and are very sweet. The plantain is only exported as flour. In the tropics it forms

a part of nearly every meal, generally cooked or made into a paste and baked. As flour it is called "gongotee." Analyses of dried and fresh fruit, flour, peels, and relative pieces of the different forms are given in detail.

D. S. PRATT.

The Determination of Tartaric Acid in Apples, Pears, Ciders and Perries. G. WARCOLLIER. Ann. fals., 4, 485.—The French law permits the addition of citric or tartaric acid to ciders or perries in amts. not exceeding 500 mg. per 1. W. has applied the best known quant. methods for tartaric acid to pure ciders and perries before and after the addition of definit amts. of the acid. The methods of Berthelot and Fleurieu, Pasteur and Reboul, Mestrezat (Compt. rend., July 16, 1906) and Muttelet (C. A., 3, 2716) were found unsatisfactory. The first named is the official bitartrate method. By its use from 232-512 mg. tartaric acid per 1. were found in pure ciders and perries. The addition of from 200-1000 mg. of the acid to the samples did not materially increase the amt. recovered. Numerous sources of error in this method are pointed out. The method of Kling (C. A., 4, 1590) was found to be entirely satisfactory. As a result of his detns. by this method the author concludes that apples, pears, ciders and perries do not naturally contain tartaric acid.

P. B. Dunbar.

Official Methods of Evaluation of Products Containing Sugar and Alcohol. F. FREYER. Vienna. Arch. Chem. Mikros., 4, 245-60.—The methods for detg. sugars by polarization and Fehling's soln. are given, with details for inversion and computation. Special methods are used with material like candied fruits that are not completely sol. in water. Candy, chocolate, candied fruits, compotes, marmalade, syrups, liqueurs, glycerol soap and condensed milk are briefly discussed. A new method for detg. alc. in perfumes, essences, etc., is given. In these cases it is necessary to remove the ethereal oil, ether, and other vol. compds. before distilling off the alc. For this purpose dilute 50 cc. of material with 50 cc. H<sub>2</sub>O in a sep. funnel, extract 3 times with 25 cc. CCl, and wash the united portions of CCl, 3 times with 15 cc. H<sub>2</sub>O to remove traces of alc. Add wash H<sub>2</sub>O to the original soln., NaCl to satn. and shake out with 25 cc. CCl<sub>4</sub>. When clear the aq. soln. is distilled and alc. detd. as usual. With essence very high in ext. it is better to distil direct and proceed as above with distillate. Alc. in acetic ether is detd. by adding 15 g. of NaOH in 100 cc. H<sub>2</sub>O to 25 g. of the ester in a distilling flask cooled by cold H<sub>2</sub>O. With frequent shaking the saponification is completed as shown by the complete disappearance of the odor. Litmus is added and nearly enough dil. H, SO, to neutralize the NaOH. The alc. is distilled off and detd. as usual. 88 g. of ester give 46 g. EtOH. The difference is due to free alc. in the original ester. D. S. PRATT.

Colorimetric Determination of Copper in Preserves. H. Serger. Chem. Ztg., 35, 935.—Lakus' electrolytic deta. of Cu in preserves (C. A., 5, 3100) is satisfactory, but too lengthy, requires too much use of Pt dishes for routine work, and is of questionable precision for small amts. of Cu. S. drains the preserves on a sieve, weighs 100 g. of the finely divided material on a filter paper placed in a porcelain dish, dries at 110°, and ignites in a quartz crucible. The ash is dissolved in 10 cc. HCl, diluted, and NH<sub>4</sub>OH added in excess. After standing a short time the soln. is filtered into a 100 cc. cylinder, and washed to a vol. of 100 cc. To a similar cylinder, containing 5 cc. NH<sub>4</sub>OH and 80 cc. H<sub>2</sub>O, is added with frequent shaking, a 0.5% CuSO<sub>4</sub> soln. until the depth of color is matched. Detas. on peas and spinach show this method accurate and rapid. There was no trouble from the NH<sub>4</sub>OH ppt. retaining Cu as noted by Graff (C. A., 3, 339).

Characterization of Artificial Permitted Colors. L. RONNET. Reims. Ann. fals., 4, 474.—A brief table of reactions for the rapid detn. of types and classes of dyes.

D. S. Pratt.

Opinions of the Kgl. Preussischen Wissenschaftlichen Deputation für das Medizinalwesen Concerning Benzoic Acid. Z. öffent. Chem., 17, 314-6.—Investigations of the BzOH question are reviewed.

D. S. P.

A Simple Method for the Qualitative Detection of Caffeine, Salicylic and Benzoic A. NESTLER. Arch. Chem. Mikros., 4, 225-31. - A sublimation method previously used by the author for the detection of extracted tea leaves in tea was applied to the detection of caffeine. The sample under exam. is pulverized, placed on a watch glass which is covered with a flat glass plate on which are a few drops of water, and subjected to a gentle heat; when casseine is present, it appears on the cover glass in needles. This method is rapid, and requires a very small sample. It is useful for the exam. of so-called caffeine-free coffee. One-quarter of a bean of this material yields only a few needles, while a very small piece of ordinary roasted coffee gives numerous needles. Kola preps. and cereal and malt coffees may also be conveniently examd. by this means. In the last case drops of fat appear on the cover glass, but no needles. The fat drops are dissolved in a little benzene, and on evapn. caffeine needles are deposited. As caffeine is not volatil with steam, the above method may be applied to the residue from beverages after evapn. A similar process may be applied to the detection of BzOH and salicylic acid. 2 or 3 drops of the soln. under exam. are placed on the watch glass, acidified with phosphoric acid and warmed until drops appear on the cover glass. These are allowed to evap, spontaneously when characteristic crystals of BzOH or salicylic acid appear. As low as 0.01% of these acids may be detected. The author has applied it to milk, strawberry and apricot marmalade, wine, vinegar, butter, and raspberry sirup.

The Determination of Formic Acid in Food. II. HEINRICH FINCKE. Nahrungsml. Untersuchungsanstalt Cöln. Z. Nahr. Genussm., 22, 88-104; cf. C. A., 5, 1300.—The object of the investigation was to study the dist. of very small quantities of CH<sub>2</sub>O<sub>2</sub> with steam, to det. the sources of error in the HgCl<sub>2</sub> method and to find a method for detg. CH<sub>2</sub>O<sub>2</sub> in AcOH. The limits of error in the HgCl<sub>2</sub> method are very wide when the amt. of CH<sub>2</sub>O<sub>2</sub> is small (2-3 mg.) owing to variations in wt. resulting from weighing the crucible containing the pptd. HgCl in the air. The error may be reduced to ±0.1 mg. by substituting for the crucible a tared 4 or 5 cm. filter which has been dried for 2 hrs. in a water bath and 6-12 hrs. in a desiccator over  $P_2O_4$ . The paper is enclosed in a weighing bottle, and great care is taken to obtain uniform conditions during weighings. When such small amts. of CH<sub>2</sub>O<sub>2</sub> are present, the vol. of soln. is kept at about 25 cc. and only 0.2 g. AcONa and 2 cc. 10% HgCl, are added. In dist. CH<sub>2</sub>O<sub>2</sub> with steam the best recovery is obtained when the vol. of the soln. is low. The recovery obtained by dist. 100 cc. containing 60 mg. CH<sub>2</sub>O<sub>2</sub> was 92.4% while 600 cc. of soln. containing 60 mg. CH<sub>2</sub>O<sub>2</sub> yielded only 37.2%. A very extended study was made of the volatil organic acids which may interfere with the HgCl, detn., and methods for removing them are suggested. In detg. CH<sub>2</sub>O<sub>2</sub> in vinegar, AcOH, etc., the acidity of the sample under exam. may be nearly neutralized with Na<sub>2</sub>CO<sub>2</sub> or CaCO<sub>3</sub> before dist. without vitiating the results. Of 9 samples of dist. vinegar examd. only 3 contained CH<sub>2</sub>O<sub>2</sub>, the max. being 0.38 parts per 1000 of total acid; CH<sub>2</sub>O<sub>2</sub> was found in all wine vinegars, max. 0.51 parts per 1000 of acid; in AcOH from wood the average was 4.75 and the max. 12.04 parts CH<sub>2</sub>O<sub>2</sub> per 1000. P. B. DUNBAR.

Maize Studies. T. F. Hanauser. Arch. Chem. Mikros., 4, 213-24.—The microscopic structure of the tissues and starch of sweet or sugar corn is presented in detail. Starch grains of different varieties of corn are also described. Good drawings accompany the text.

A. W. Broomell.

Identification of Olive Seeds in Pepper. C. V. GAROLA AND V. BRAUN. Ann.

jals., 4, 467.—The well known method (*Ibid.*, 36) of distinguishing the adulterant by coloring it with p-phenylenediamine-HCl has been improved by the authors. The powder, on a microscopic slide, is moistened with a 1% soln. of the reagent. After 15 min., an exam. is made, using the polarizer. The pepper appears but indistinctly, while the olive seeds colored reddish yellow, show plainly. Photographs are best made by staining the materials with a soln. of I in KI, when the adulterant alone shows white with polarized light. The % of adulterant may be estd. and as low as 5% identified.

A. W. BROOMELL.

Definition of Vinegar. T. SCHUMACHER. Z. Nahr. Genussm., 22, 104.—The following provisional definitions were laid before the Freien Ver. deut. Nahrungsmittelchemiker to serve as a basis for discussion. Vinegar is the product derived by the acetic fermentation of alc. liquids, and is named after the raw material from which it is made—as wine-, beer-, malt-, or honey-vinegar—but the products so named must be derived exclusively from the source indicated. Preps. designated "diluted" wine-, malt-, honey-, etc., vinegar may be sold as such provided they contain not less than 20% by vol. of the raw material indicated, the finished product to contain not less than 5% AcOH. Vinegar substitutes or "essences" are made by liberating AcOH from acetates. These essences should contain not less than 60% AcOH. The minimum amts. of AcOH permitted in vinegar are: vinegar (without qualification) or "table" vinegar 3.5%, "preserving" vinegar 5%, "double" vinegar 7.0%, and "spirits" of vinegar 10.5%. Other preps., made by dilg. AcOH or vinegar essences must contain not less than 3.5% and not more than 15% AcOH, the label plainly to indicate their origin and prevent confusion with fermentation vinegars. The sale of vinegar containing preservatives or NaCl is prohibited unless their presence is stated on the label. Artificial ethers, perfumes or colors (excepting caramel), organic or inorganic acids when used in vinegar are regarded as adulterants. Vinegar infested with growths of various kinds, vinegar eels or moulds, likewise those containing Pb, Zn or Cu are classed as unfit for food. A. F. SEEKER.

A Paraffin Lined Vinegar Cistern. H. B. Bonebright. Pure Products, 7, 338-9.

—It was found that a cement cistern well lined with paraffin makes an ideal receptacle for vinegar. The product thus stored is equal if not superior to vinegar stored in wooden barrels.

C. P. Wilson.

Improvers. Bonis. Ann. fals., 4, 461-7.—This name is given to all sorts of flavorings and bouquet producers. It is recommended that some natural vegetable flavors be allowed in commerce but that all artificial esters and flavors or bouquet producers of that class be entirely prohibited from entering the trade.

C. P. W.

Ginger Extract. JOHN PHILLIPS STREET AND C. B. MORISON. U. S. Dept. Agr., Bur. Chem., Bull. 137, 76-9.—Attention is called to the high alc. and low water solubility of the solids occurring in ginger exts. and the variations when the exts. have been prepared with dil. alc. Winton's Pb no. as applied to vanilla exts. was tried as a means of detecting adulteration but was not found available. Analyses of genuin and compd. exts. are shown and the methods used for detg. alc., total solids, solids sol. in alc. and solids sol. in water are given.

E. M. CHACE.

Report of the Associate Referee of the A. O. A. C. on Flavoring Extracts for the Year 1910. E. M. CHACE. U. S. Dept. Agr., Bur. Chem., Bull. 137, 64-76.—The collaborative work for the year was confined to trying out the available methods for the detn. of citral in lemon oil. The colorimetric methods tried were based on the reactions between citral and fuchsin-sulfite solns. and between citral and m-phenylene-diamine. The volumetric methods tried were those of Bennett (C. A., 3, 942), de-

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pending on the reaction with hydroxylamine and an unpublished method of Klebers, depending on the phenylhydrazine reaction. The reports from analysts were highly favorable to the Kleber method and it was adopted as a provisional method of the Assoc. Bennett's method gave results equally accurate but, owing to the difficulty in detg. the end point, was not recommended for adoption. Owing to the great multiplication of the individual error, the colorimetric methods were unsatisfactory. Other important changes in, and additions to, the provisional methods included a change in the vanillin and coumarin method, in the color tests on lemon extracts and the addition of methods for almond, cassia, cinnamon, clove, ginger and peppermint extracts. The methods for caramel, capsicum, nutmeg and wintergreen were left for further study.

A New Characteristic of Almond Coffee, Cyperus esculentus. F. NETOLITZKY. Arch. Chem. Mikros., 4, 260-2.—At present the term "almond coffee" is applied to a whole series of substitutes, although it originally meant the roasted and pulverized root stalks, tubers, of Cyperus esculentus. It is much used in South Europe and North Africa as a food material. The earth almond played an important role in food in ancient Egypt, and the author identified it in the urins from prehistoric graves in Niltali. In the contents of the corpses it was found only by ashing the preparations and utilizing the characteristic siliceous cell structure. This is also the best means of identification in strongly roasted substitutes of the almond. The method is new, and is carried out by ashing the material at as low a temp. as possible. Mount in H<sub>2</sub>O and acidify. The siliceous cells so obtained are polygonal and tend to large groups. In each cell are 2-6 ball-like knobs making the cell not unlike dominoes, and which serve for recognition.

Examination of Tea. I. A. A. BESSON. Chem. Ztg., 35, 813-5.—Many samples of tea from authentic sources were examined. The % of leaves to stems was detd. by hand sorting after b. for 15 min, and floating in a dish. The fractions thus obtained were dried and weighed. This relation seems to depend upon the source of the tea and is not a criterion of the value of the sample. The ash with most teas is greenish due to the Mn content. Only with green tea is this lacking, due to absence of alkali, as with added soda the green ash is obtained. In like manner the ash from green tea does not dissolve with red color in acid, while that from black tea does. Whether this is the result of bacterial action is not known. Various tables of analyses are given. (II). Ibid., 830-2. The ash of stems and leaves of completely extracted tea gives the mineral content of these and, lacking alkali, is never green. The stems contain more water sol. inorganic material than the leaves, but the latter more organic. Tables of many varieties of teas are given showing ash, color, and alkalinity. Caffeine was detd. by the method of Keller, and tannin by that of Fleck. Tasting tests were also made. Often the most satisfactory samples of tea contain a high % of stems, and that condemning on this basis is without foundation. D. S. PRATT.

Comparative Study of the Usual Methods for the Determination of Nicotine in Tobacco Extracts. J. Toth. Chem. Ztg.; 35, 926-7.—The author gives in tabular form results of nicotine detns. on 31 samples of tobacco ext. by his own method and those of Kissling, Ulex, and Degrazia. Kissling's method is not applicable in the presence of pyridine bases and takes too long to be of much commercial value. The method of Ulex gives results higher than those of any other method. In another table the methods of Toth and Kissling are compared with Koenig's method and the authors modification of the same, which consists in using xylene in place of toluene as a solvent for the nicotine. The polarimetric method using xylene as the solvent is the preferred method.

C. P. Wilson.

Feeding Experiments with Tilletia. A. SCHEUNERT AND E. LÖTSCH. Dresden. Z. Infektionsk. parasit. Krankl. Hyg. Haustiere, 9, 177-87; Chem. Zentr., 1911, I, 1714.—The widespread opinion that feed containing spores of Tilletia produces serious illness to cattle is ungrounded. Swine suffered no injury, the food was not rejected, and the swine bore well-developed young.

M. X. SULLIVAN.

The Soy Bean (WAERDEN). 27.

Properties of Human Milk (POLENAAR, FILIPPO). 11.

Digestibility of Potato Syrup (HAMBURGER). 13.

- U. S., 999,972, Aug. 8. M. EKENBERG, London, Eng. Assignor to Techno-Chem. Lab., Ltd., same place. Desiccating milk by spraying it into a heated mixt. of CO<sub>2</sub> 6 and steam 1 part by vol. The product is in the form of minute spherules containing absorbed CO<sub>2</sub>.
- U. S., 1,000,041, Aug. 8. J. H. SCHNEIDER, Cincinnati, Ohio. Preserving corn cut from the cob by mixing with sugar, salt and hot H<sub>2</sub>O in the proportions of 5 gals. of cut corn, 2.5 lbs. sugar, 2.25 lbs. salt and 5 qts. of H<sub>2</sub>O, b. the mixt. for not less than 20 min. and then sealing in air-tight vessels.
- U. S., 1,000,239, Aug. 8. C. W. Dayton, Chatsworth, Cal. Ripening extracted honey by confining a body of honey from contact with the atm. except at 1 surface, interposing a moisture-absorbing barrier, e. g., muslin, between this surface and the atm. so as to leave a space between the surface and the barrier to receive the gases given off from the honey and air admitted slowly through the barrier, thus maintaining a slowly changing and relatively still body of air next the surface of the honey.
- U. S., 1,000,240, Aug. 8. *Idem*. Curing and preserving honey by placing it in a closed chamber, supplying hot air to the interior of the chamber above the level of the honey, continuing the supply of hot air until the atm. within the chamber is at a temp. of 21-32° and all exuding moisture is evapd. from the surface of the honey, inducing a downdraft of the moisture-laden air to exhaust it from the chamber below the level of the honey, and repeating this course of treatment periodically,  $\epsilon$ . g., once every 24 or 48 hrs., until the honey ceases to ferment.
- U. S., 1,000,409, Aug. 15. F. W. Howe, Framington, Mass. Preparing milk for delivery by filling the containers with fresh milk, subjecting to a vacuum until the gases and animal heat have been removed from the milk, and then sealing without access of atmospheric air.
- U. S., 1,000,433, Aug. 15. J. B. MOSZCZENSKI, New York, N. Y. Assignor to Tartar Chem. Co., Jersey City, N. J. Making cream of tartar by treating crude material containing K bitartrate with CaO to form K tartrate soln. and Ca tartrate, treating the latter with H<sub>2</sub>SO<sub>4</sub> to produce tartaric acid, bleaching the tartrate and tartaric acid solns. and combining them to ppt. cream of tartar.
- U. S., 1,000,641, Aug. 15. A. TEBBIT, Medina, Ohio. Machine for homogenizing milk.
- U. S., 1,000,686, Aug. 15. L. C. MERRELL, Syracuse, N. Y. Assignor to Merrell-Soule Co., Syracuse, N. Y. Acid milk-powder wholly sol. in H<sub>2</sub>O and containing 1.25-3.5% lactic acid.
- U. S., 1,000,692, Aug. 15. L. ROSELIUS, Bremen, Ger. Removing caffeine from roasted coffee by moistening the coffee, partially drying it, treating it with a soln., e. g., NaCl, to facilitate electrolytic action, decomposing the salts of caffeine in the

beans by electrolysis, extracting the caffeine by a solvent, e. g., benzine and alc., which does not injure the other constituents of the coffee, subjecting the coffee to dry steam to remove the residual traces of solvent and heating the coffee to dry it.

U. S., 1,000,931, Aug. 15. L. C. MERRELL, I. S. MERRELL and W. B. GERE, Syracuse, N. Y. Apparatus for desiccating milk, etc.

# 13. NUTRITION.

WM. J. GIES. NORMAL.

Metabolic and Respiratory Investigations Concerning Protein Feeding. A. MUELLER. I. med. Klinik, Wien. Zentr. ges. Physiol. Path. Stoffw., 5, 617-29.—In normal man, when a considerable N retention is obtained by introduction of much protein and many calories, the increase in O consumption does not occur in proportion. This is due to an accumulation of reserve protein as well as to the protein intake. The sudden transition from a chronic state of poor nourishment, and loss in respiratory exchange, to a normal nourishment and N retention is accompanied by a rapid increase of O consumed. This increase occurs with the N retention but does not continue proportionally with it. It is therefore no direct indication of gain in flesh, but of an increase in the decomp. of the protoplasm.

C. A. Brautlecht.

Biological Behavior of Potassium. E. BIERNACKI. Inst. allgem. expt. Path., Lemberg. Zentr. ges. Physiol. Path. Stoffw., 6, 401-7.—From previous expts. on dogs (cf. C. A., 4, 796; 5, 548, 3078), B. stated that with increase in body wt. there was an increase in K excretion, and under the same conditions there was a retention of Na and Ca. In this article the previous statements are confirmed. Larger doses of NaCl also diminished N excretion, especially with a high meat diet. Rice, which was the carbohydrate portion of the diet, probably caused a retention of some K. The last statement agrees with observations on carbohydrate metabolism and with the nature of most vegetables or high carbohydrate foods which naturally have a high content of K. The N of the feces was unchanged when either NaCl or KCl was used in the diet; their water content was greater with the KCl diet. Large doses of NaCl in the diet decreased the Ca excretion and increased that of K.

C. A. BRAUTLECHT.

Some Questions of Metabolism and Nutrition. A. GIGON. Münch. med. Wochschr., 58, 1343-7.—G. carried out expts. with the Tigerstedt and with Jaquet's respiration chambers, and with Jacquet's spirometer, and obtained results that agree well. Having obtained the respiratory quotient for a fasting period, he detd. it for varying periods after the eating of weighed amts. of casein. He concludes that the larger part of C remaining after formation of urea is converted into fat and a smaller part goes to form carbohydrate.

G. R. Henry.

The Adaptability of the Animal Organism to Superabundant Food Supply. E. GRAFE AND D. GRAHAM. Med. Klin., Heidelberg. Z. physiol. Chem., 73, 1-67.—The object of the investigation was to det. whether the animal organism can adapt itself, without increase of body wt., to a diet not rich in protein but otherwise much in excess of its requirement; and, if it can, in what way the adaptation takes place. The subject was a bitch of 20 kg., and the expt. lasted 107 days. Liquid and solid ingesta and excreta were subjected throughout to exact chem. and calorimetrical analysis, and at intervals of a few days respiration expts. of 7-24 hrs. duration were carried out. The whole expt. fell into 7 periods. In the 1st (21 days) food was withheld; the av.

daily loss of N was 3.975 g.; the wt. fell to 15 kg.; by respiration expts. the minimal food requirement was detd. to be 822.8 cal. for a wt. of 20 kg. In the 2nd (7 days) the av. daily intake was 2243.9 cal. = 280% of the requirement, with 17.177 g. N; N was retained at the rate of 12.601 g. daily and the body wt. returned to normal. In the 3rd, 4th, and 5th periods (of 29, 11, and 19 days) the intake was respectively 300, 200, and 130% of the requirement, with an av. N content of 19.68, 15.217, and 14.15 g. daily; but in spite of the excessive caloric value of the diet, and of the fact that during these 59 days 298.1 g. N were retained in the body, the wt. of the animal remained practically const. at 20 kg. In the 6th period (10 days) only the maintenance requirement was given, and in the 7th the animal was starved for 7 days; the wt. fell 1 and 1.5 kg. The failure of the enormous over-feeding of the middle 3 periods to produce increase of body wt. could not be explained by altered motility or by compensatory loss of H<sub>2</sub>O; it could be accounted for only by an increase of the oxidation within the body. Respiration expts. carried out 30-36 hrs. after meals showed that during the period of over-feeding the fasting metabolism was actually increased by about 40%. The existence of a "luxus consumption" is thus for the first time directly proved. The over-abundant supply raised considerably the food requirement. Respiration expts. made immediately after meals indicate that during the continuance of overnutrition the combustion of the foodstuffs becomes more and more intense. In these ways the adaptability of the organism is manifested. An abs. equil. between food supply and heat production was not attained, since at the end of the period of excessive feeding a slight retention of N still existed. The part played by luxus consumption of the kind described may be of very different importance in different individuals; this may account for the great variations exhibited by both adult and growing organisms in their capacity for fattening. A. HUNTER.

The Stimulation of the Gastric Secretion under the Influence of Water Drinking with Meals. F. WILLS AND P. B. HAWK. Proc. Am. Physiol. Soc., 1910, 32; Am. J. Physiol., 27.—Under the influence of H<sub>2</sub>O drinking at meals by normal men the NH<sub>2</sub> content of the urin is increased. This increase is considered an indirect index of the activity of the gastric glands.

J. F. L.

Losses and Regeneration in Life's Process. Max Rubner. Berlin. Sits. kgl. presss. Akad. Wiss., 1911, 440-57; through Chem. Zentr., 1911, I, 1598.—It is not possible to replace the minimum protein requirement by other foodstuffs. The constant loss of N is due to the continual breaking down of cells (loss of hair, epidermis, mucous, and cellular secretions) as well as the destruction of blood. All these losses taken together are the "wear and tear." The % "w. and t." divided by the energy consumed is the "w. and t." quotient. Divergent values for the minimal protein consumption found in literature can be explained by the fact that each protein possesses definit replacing values. Expts. on dogs, birds and man have fixed the "quotient" for the smallest % protein involved in the general metabolism at 4%. According to Thomas, the "quotient" calc. from the urinary N in man is 30 mg. N per kg. body wt. or about 1/1000 of the total body N per day. The fecal N is derived mainly from the remains of the digestive secretions. Loss of N due to muscular activity is independent of the organ activities. Each 100 kg. cal. muscular activity causes a loss of about 41 mg. N. Organic functions must utilize more N than muscular activity.

G. M. MEYER.

The Perfecting of Protein Nutrition. José R. CARRACIDO. Anales soc. españ. fis. quim., 8, 349-55; Rev. real acad. cien., Madrid, 9, 212-8.—In discussing the large ingestion of protein material as compared with the amt. assimilated by the human organism, as well as the danger of auto-intoxication incident thereto, the author ad-

vocates a more rational system of dietetics in which a combination of protein foods could be so arranged (according to the kind and proportion of pre-formed amino acids contained in them) as to qualitatively and quantitatively furnish the amino acids of the blood plasma proteins (insofar as the necessary analytical data may be known). For instance, gelatin would compensate the deficiency of glycocoll and the excess of tyrosine and glutaminic acid due to a milk diet, etc.

H. S. Paine.

A Method for Determining the Digestibility of Potato Syrup. H. J. HAMBURGER. Chem. Weekblad, 8, 615-20.—The digestibility of potato syrup as compared with cane or beet sugar is proportional to the amts. of reducing sugars after inversion which may be obtained from the same quantity of each substance. This was detd. by fermentation with yeast and measuring the CO<sub>2</sub> produced. 100 g. of potato syrup have a food value equiv. to 61-70 g. of cane or beet sugar. The dextrin of potato syrup has a beneficial action on the chyle, thereby aiding digestion.

C. P. WILSON.

#### ABNORMAL.

Digestion of Meat in the Dog Stomach under the Influence of Opium Tincture Containing or free from Morphine. A. Delcorde. Inst. Therap., Univ. Bruxelles. Intern. Beitr. Path. Therap., 3, 24-52.—Under the influence of opium tincture the stomach of a dog retained raw or cooked meat for a longer time. The chyme began to pass into the duodenum 4-6 hrs. after the feeding, the stomach requiring about 3 times as long to be emptied. When opium is given, protein hydrolysis, compared with the normal, is more nearly complete in the fundus than in the pyloric region. Constituents of opium other than morphine caused a retention of meat in the stomach and also-constipation (cf. C. A., 5, 2492).

Experimental Studies of Injected Nourishment. Wasserthal. Exp.-Biol. Abteilung d. Königl. Path., Inst. Univ. Berlin. Intern. Beitr. Path. Therap., 3, 111-6.—Injections of milk, alc., egg-yolk, Liebig's ext., Witte's peptone, salt, sugar; etc., in water in various cones. were found to temporarily stimulate gastric secretion. The degree of stimulation depended on the comp. and may have been caused reflexively as well as through the blood.

C. A. Brautlecht.

The Hygienic Significance of Alcoholic Liquors. R. ROSEMANN. Z. Nahr. Genussm., 22, 24-34.—Alc. is quickly absorbed in the body, and all but 2% burned. Its chem. energy (1 g. alc. = 7 Cal.) is utilized as is that of a food, although practically its food value is small in consideration of its other effects. It dulls sensations and tends thus to disarrange the mechanism of the vital functions of the body; hunger is arrested with danger from malnutrition; the feeling of cold is arrested with danger of abnormally lowering body temp.; fatigue is arrested, with danger of overexertion and later enervation. Harm to the sound growth of the body from moderate alc. drinking has not been proved. Alc. is a very effective and therefore very dangerous, but when rightly applied, very valuable food.

A. W. Broomell.

Metabolism Experiments with Albulactin on Artificially Nourished Infants. Karl, Bornstein. Sauglingsheim. Leipzig-Connewitz. Arch. Kinderh., 56, 16-26.—A baby 4 mos. old received daily 900 cc. milk (6 × 150 cc.) 1: 1 water. This continued for 12 days. Then for 12 days 6 g. albulactin (a prepared sol. lactalbumin) was added to the usual diet. The N excretion varied but little, showing a marked retention. Likewise the stools though thin showed marked reduction of solid substance. In spite of N retention, the body wt. did not increase. This was due to loss of water only, resulting merely in a body consolidation. The Ca excretion was reduced, from 0.653 to 0.473 g. of an intake of 1.08 g. The child seemed perfectly normal and happy.

Walter A. Jacobs.

The Significance of Calcium in the Pathology of Rickets. IV. Phosphorus Metabolism in Rickets. J. A. SHABAD. St. Petersburg. Arch. Kinderh., 54, 83-109.—In the developing stage of rickets the excretion of both CaO and P<sub>2</sub>O<sub>4</sub> is increased. The increased P.O. excretion most often exceeds the equiv. CaO content of the bone tissue so that apparently the nervous tissue participates in the pathological process. The increase in the P excretion proceeds exclusively in the feces, whereas the P content of the urin falls below the normal (hypophosphaturia). The ratio between the P content of the urin and feces of infants is normally 80: 20, becoming in rickets 65: 35. In artificially nourished infants and in older children the normal ratio of 60: 40 is reversed and becomes 40-48: 60-56. During convalescence the total P excretion is lower than normal; the ratio returns to normal, whereby a relative increase in the excretion of P in the urin (75: 25) is observed (hyperphosphaturia). A close relationship between the Ca and P excretion in the feces was observed. Increase in the fecal Ca led to P retention in the intestin and vice versa. Since in rickets more P than Ca is removed, the possibility is excluded of referring the P excretion to the increased Ca elimination. It is more probable that the increased P excretion is of primary significance in the disturbance of metabolism. W. A. J.

## 14. WATER, SEWAGE AND SANITATION.

#### EDWARD BARTOW.

Rapid Detection of Nitrates and Nitrites in Water by means of a New Reagent—Hydrostrychnine. G. Denigès. Bull. soc. chim., 9, 644-6.—D. has recently recommended this reagent (C. A., 5, 2792) for the detection of Br. If 0.5 cc. are added to 10 cc. of a water containing nitrites, a rose color immediately develops. Comparison with NaNO, standards treated the same way gives quant. results. Nitrates give a similar color with this reagent providing an amt. of conc. H<sub>2</sub>SO<sub>4</sub> equal to 0.5 the vol. of the water used, is added. If both salts are present it is either necessary to run a blank using an amt. of NaNO<sub>2</sub> equiv. to that found or to remove the nitrites. This may be accomplished by evapg. with a few drops of NH<sub>4</sub>OH and AcOH. Chlorides do not interfere.

W. F. Langeljer.

Action of Seltzer Water on Lead, Tin and Antimony and Causes of Intoxication by Chemical Alteration. A. Barillé. Compt. rend., 153, 351-3.—A study of the action of carbonated water upon the various metals used for stopping the siphons. The metals used are either pure Sn or an alloy of Sn with Pb or Sb. The alloys are effected much more readily than pure metals. There is little difference in the amt. dissolved after 6 months' action, between alloys containing only 0.5% and those containing 40% of Pb. The soln. of Sb in carbonated waters is also assisted by electrolytic action. Only glass should come in contact with carbonated waters. If metal be used, it should be covered with some impervious cement or varnish. Much smaller amts. of Pb than those usually considered harmless will cause serious bodily derangements, after long-continued use.

Misbranding of Mineral Waters—"California Waters of Life." Notice of Judgment No. 830, Food and Drugs Act, 1911.—Analysis showed no ingredients possessing therapeutic value superior to those of ordinary spring waters. Claims not justified.

E. Barrow.

Purification of Water. H. ACKERT. Papier Fabrikant, 9, 1128-32.—In mfg. most kinds of paper a clear, colorless water, free from organic substance is required. In purifying water the following points are recommended: Avoid boiler compds.

made according to a secret formula; use lime and soda in the special installation; avoid a lime saturator, using milk of lime; obtain reliable app. for distributing the chemicals in proportion to the raw water; use as large settling tanks as possible, arranged so that velocity of water may be as low as possible.

E. Bartow.

Study of the Water of Parc Sainte-Marie (Source Lauternier). A. GAUTIER AND C. MOUREU. J. pharm. chim., 3, 281-6, 335-41, 391-8.—Besides the ordinary constituents bromides, iodides, fluorides, borates, Sb, Sn, As, Ra, A, Xe, Kr, He, and N were found in traces. The water is nearly free from bacteria. V. K. CHESNUT.

Purification Experiments with a Surface Water in Australia. HAROLD WASTENEYS.

New England W. W. Assn., 1911; Eng. Record, 64, 324-5.—A brief review of expts. in handling a water from a reservoir full of fresh water plants, particularly blue-green algae. The object was to remove odos, taste, color and Fe. Mechanical filtration with coagulants was not successful. Slow sand filters were not altogether satisfactory in summer. Intermittent filtration was recommended.

LANGDON PEARSE.

Emergency Artesian Well System at St. Paul, Minn. Anon. Eng. Record, 64, 306.—To relieve a threatened water shortage, six 12-in. artesian wells were driven to a depth of 700 ft. The efficiency developed (total ft. lbs. of water delivered + total foot lbs. steam consumed) was 4.9%. The plant in the test delivered 6,350,000, gals.

Operating Results of the Nashville Purification Works. Anon. Eng. Record, 64, 337.—Cumberland River water was treated with Al<sub>2</sub>(SO<sub>4</sub>)<sub>2</sub> stored 1 3/4 days, 0.1 grains per gal. of Ca(OCl)<sub>2</sub> added and stored 1 3/4 days longer. The turbidity was practically removed, with a reduction of bacteria to an av. of 97 per cc. The cost was \$2.87 per mil. gals. for chemicals and labor, and 25c. additional for cleaning reservoir. The amt. of Al<sub>2</sub>(SO<sub>4</sub>)<sub>6</sub> averaged 1.29 grains per gal., with a min. of 0.8 and a max. of 1.6.

L. P.

Operation of New Orleans Filters. John L. Porter. Eng. Record, 64, 356.—During 1910, the chemicals added averaged: CaO, 4.8 grains per gal., and FeSO<sub>4</sub>, 0.76 grains per gal. Bacterial results were good. No positive B. coli test occurred in the filtered water. The filter runs averaged 136.6 hrs., and the % of wash water 0.75. The net rate of filtration averaged 59.9 mil. gals. per acre per 24 hrs. Cost of chemicals, supervision, labor, etc. = \$6.60 per mil. gals. Cost of wash water and cleaning reservoirs raises this to \$6.77.

Administration of Cleveland Water Works during 1910. L. C. Smith. Eng. Record, 64, 357.

L. P.

An Emergency in the Operation of an Iron Removal Plant. Anon. Eng. Record, 64, 375, 379.—The water company, in an emergency, used Al<sub>2</sub>(SO<sub>4</sub>)<sub>8</sub> instead of lime. The addition of 2 grains per gal. CaO and 2 Al<sub>2</sub>(SO<sub>4</sub>)<sub>8</sub> gave the best results, as CO<sub>8</sub> present in the water interfered with the formation of a floc unless CaO were added.

L. P.

The Municipal Water Purification Plant at Grand Rapids. J. W. Armstrong. Eng. Record, 64, 379-81.—The details for handling the CaO, Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> and "hypo" are novel. A concrete mixer is installed for slaking the lime. The "hypo" is to be handled under water.

L. P.

Water Purification Works at Toledo, Ohio. D. H. GOODWILLIE. Eng. News, 66, 396-7.

J. J. M.

Reinforced Concrete vs. Vitrified Clay Pipe for the Roswell Park Pipe Line. Z. VAUGHN. Eng. News, 66, 379.—Polemical against B. Brooks; C. A., 5, 3140.

J. J. M.

Report on Sewage Disposal Investigations. Studies of the Philadelphia Bureau of Surveys. Sci. Am., 72, 11; J. Soc. Chem. Ind., 30, 976.

J. J. M.

Chicago Tests of Emscher Tank. J. Assn. Eng. Soc., 47, 37-41; Mun. J. Eng., 31, 291.—A brief discussion of the working of the Emscher tank. The removal of suspended matter ranges up to 65%. The sludge accumulation has been at a max. rate of 2 cu. yd. per mil. gals. of sewage, 90% water. Sep. sludge digestion is not recommended. No H<sub>2</sub>S odor has been noted. Reference is made to a sewage disposal plant at Kings Park, N. Y., where freshly settled sludge is run into covered trenches.

L. P.

Imhoff Sewage Tanks at Winters, Cal. Eng. Record, 64, 360-1.

L. P.

Trials of Sewage Precipitants at Wakefield. J. P. WAKEFORD. Roy. Sanitary Inst. Eng. Record, 64, 355.—The Wakefield sewage contains industrial wastes. Chem. pptn. is followed by percolating filters. The precipitants tried were (1) CaO, (2) CaO and  $Fe_2(SO_4)_3$ , produced by oxidizing copperas soln., (3) CaO and copperas, (4) CaO and  $Fe_2(SO_4)_3$  in slabs, (5) CaO and  $Fe_2(SO_4)_3$  produced by dissolving  $Fe(OH)_3$  in  $H_2SO_4$ . The conclusion was that lime followed by  $Fe_2(SO_4)_3$  in soln. was most effective, and that dissolving  $Fe(OH)_3$  in  $H_2SO_4$  is cheaper than oxidizing copperas soln.

L. P.

Temporary Screening Plant at Flushing, N. Y. Eng. Record, 64, 303. L. P.

Production of Goiter (BIRCHER). 11.

U. S., 1,000,608, Aug. 15. J. W. Kenevel, Philadelphia, Pa. Electrolytic apparatus (for purifying water) comprizing a chamber with oppositly disposed inlet and outlet pipes, several groups of electrodes in the chamber each of which include coacting parallel plates, the plates of the different groups being staggered, several hoods for the chamber and connections by which each of the hoods supports one of the groups of plates.

### 15. SOILS AND FERTILIZERS.

#### F. P. VEITCH.

Agricultural Chemistry. E. Blanck. Fortschritte Chem., 4, 211-29.—Review of progress in the year ending July, 1911.

J. JOHNSTON.

Determination of Sulfuric Acid in Soils. P. DE SORNAY. Intern. Sugar J., 13, 497.—Long study has shown that in the ordinary procedure we do not recover all the  $H_pSO_4$  in soil analysis. The following method has been found to give excellent results: Dry 10 g. of the soil, 1 g. KNO<sub>3</sub>, and 5 cc. of water on the sand bath, calcine, and then treat with 25 cc. of HCl. Heat on the sand bath again to complete dryness; the SiO<sub>2</sub> is rendered insol. After taking up with 5 cc. of water and 20 cc. of HCl, again heat on the sand bath for 30–45 min. without going to dryness, filter, and wash with 100–25 cc. water. To the filtrate and washings add BaCl<sub>2</sub>, heat, and conc. a little to facilitate pptn. Complete the detn. in the usual manner.

A. GIVEN.

Preliminary Investigation on the Storage of Lime Nitrogen in the Tropics, and the Reactions which Take Place thereby. C. J. Millo. Arch. Suikerind., 19, 983-1035.

—The lime N is admitted to Java (duty-free) is a gray-black powder of the following comp.: CaCN<sub>2</sub>, 50.9%; CaO, 27.2; free C, 9.5; Fe<sub>2</sub>O<sub>3</sub> + Al<sub>2</sub>O<sub>4</sub>, 1.8; SiO<sub>2</sub>, 1.9; MgO<sub>4</sub>.

0.04; CO<sub>2</sub>, SO<sub>2</sub>, OH not detd. One g. lime N treated with NaCl soln. evolved: 0.154% C<sub>2</sub>H<sub>2</sub>, 0.016 H<sub>2</sub>S, 0.004 PH<sub>3</sub>, no HCN. Exposed to a warm, moist atm. the surface layers cake and lose N. Some cyanamide is changed into dicyanamide. A sample containing, on Nov. 1, 15.62% N as cyanamide and 0.33% as dicyanamide, contained, on July 1 (having been stored in sacks in the meantime), 1.74% N as cyanamide and 11.37% as dicyanamide. Though the reactions were not established, it was detd. that on storing in a tropical climate the following products were formed: monobasic Ca cyanamide, Ca(CNNH)<sub>2</sub>; dibasic Ca cyanamide, CaCN<sub>2</sub>(OH)<sub>2</sub>.6 H<sub>2</sub>O; Ca cyanamidocarbonate, CaC<sub>2</sub>N<sub>2</sub>O<sub>2</sub>.5 H<sub>2</sub>O; cyanamide, dicyanamide, urea; unidentified N compds.; (NH<sub>4</sub>)<sub>2</sub>CO<sub>2</sub>; and NH<sub>2</sub>.

Use of Phosphate Fertilizers in the Cultivation of Wheat. ROBERTO OPAZO.

Anal. agronom., 6, 119-33.

G. A. ROUSH.

Fixation of Atmospheric Nitrogen (BECKMAN). 4. Air-nitrogen Fertilizers (MILO). 28. Fertilizers (SCHANDER). 28.

U. S., 1,000,290, Aug. 8. S. Peacock, Chicago, Ill. Assignor to Am. Cyanamid Co., New York, N. Y. Making phosphorus pentoxide and calcium aluminate from bauxite and phosphate rock in a single operation by mixing them in the proportions of 1053 and 2000 parts, resp., heating to about 1500° while forming the material into thin layers to permit the P<sub>2</sub>O<sub>4</sub> to escape and separately collecting the latter and the CaAl<sub>2</sub>O<sub>4</sub> formed.

U. S., 1,000,311, Aug. 8. F. S. WASHBURN, Baltimore, Md. Assignor to Am. Cyanamid Co., Baltimore, Md. Producing phosphorus pentoxide and cementitious material by finely dividing and mixing crude phosphates of Al and of Ca in the proportions of 1000 to 4680 parts, heating the mixt. not much above 1700° and collecting the sublimed oxide.

## 16. FERMENTED AND DISTILLED LIQUORS.

#### ROBERT WAHL.

An Instructive Case for Wine Experts. F. MALLMANN. Z. Nahr. Genussm., 22, 236-9.—Samples taken at the same time from different parts of a 1000 l. tun show very marked differences.

C. O. Dodge.

Advances in the Fermentation Industries during 1909 and 1910. P. RINCKLEBEN. Chem. 21g., 35, 993-4, 1006-8.

J. J. M.

The Determination of Volatil Acids in Wine. K. WINDISCH AND F. RORTTGEN. Kgl. Tech. Inst., Hohenheim. Z. Nahr. Genussm., 33, 155-70.—Solns. containing known amts. of AcOH, formic, BuOH and lactic acids were distilled with steam, and the recovery tabulated. The first 3 are completely volatil when 200 cc. is distilled off, provided the wine contains less than 0.2 g. acid per 100 cc., and the vol. during distillation is reduced to 50 cc. and maintained there. Not over about 200 cc. distillate should be obtained or lactic acid will be found to vitiate the results. Superheated steam is to be avoided for the same reason. Lactic acid is present in wine from bacterial action on malic acid.

D. S. Pratt.

The Nitric Acid Content of Pure Wines. J. TILLMANS. Z. Nahr. Genussm., 22, 201.—The author's method for the estn. of small quantities of HNO<sub>2</sub> in milk (C. A.,

5, 727) cannot be applied to wines without modification because the latter contain substances which destroy the diphenylamine reaction. The wine is prepared as follows: 10 cc. are treated with 0.2 cc. satd. NaCl soln. and some coarse, nitrate-free, animal charcoal and diluted 5-20 times with water. The mixt. is then evapd. almost to dryness with frequent stirring and finally diluted to the original vol. and filtered. This treatment removes the interfering substance and after adding 1 cc. glacial AcOH the quant. detn. may be carried out as previously described. A large number of pure German wines of known origin were examined, most of which contained nitrates, the max. being 18.75 mg. N<sub>2</sub>O<sub>6</sub> per 1. The estn. of nitrates is, therefore, without value for the detection of added water in wines.

The Detection of Substitutes of Spirits for Aged Whisky. A Discussion of the Chemical Data Presented in the Trial of U. S. vs. Nine.Barrels of Whisky. A. B. Adams. J. Ind. Eng. Chem., 3, 647-55.—The conclusions reached were that the presence of other spirits in a package of double stamped whisky can be proved by chemical analysis, and that analyses of samples taken from packages in the same warehouse were proper evidence against similar whisky under seizure. As regards the suspected samples they were not similar to the genuin samples in any points except proof and depth of color.

C. A. Nowak.

Some Remarks on the Practical Determination of the Color of Wort in the Brewery.

R. JUERST. Pure Products, 7, 295-8.—The regulation of the color depends on a small filter tube attached to the outlet pipe through which a clear sample of wort can be drawn direct from the wort cooler at any time and the color examd. C. P. WILSON.

The Admixture of Carmel Malt to the Malt Mash. F. Schoenfeld. Wockschr. Brau., 28, 371-4. C. A. N.

Loss of Alcohol by Evaporation in Fermenters. Forn. Z. Spiritusind., 34, 439-40.—The fact that a fermenter is only 1/2 or 3/4 filled does not minimize the loss of alc. by evapn. Covering is likewise of no value. Only by washing the escaping CO<sub>3</sub> practically all the alc. lost can be recovered. This method is profitable only on a large scale.

C. A. N.

The Biological Examination of Brew Water. J. Schlesinger. Allg. Z. Bierbr. Malsfabr., 39, 358-61.—A method is described by the author. The author's method (as compared with those of Hansen and Wichmann) gives results easier of interpretation and offers the advantages that wort and beer require only a little diln. and that a smaller number of flasks are necessary for the test. It is not intended to replace the method of Will but should be supplemented by it in framing a final judgment. C. A. N.

The Physiological Examination of Beer. Anon. Allgem. Z. Bierbr. Molzjabr., 39, 381-2.—A review; see C. A., 5, 1645, 2128, 2282, 2283, 2515 and 2883. C. A. N.

The Work of H. Eng. "On the Testing of Thujone in Absinthe." E. PHILLIPS AND BH. V. FALLENBERG. Schweiz Wochschr., 49, 418-20.—Thujone is oxidized by Ag<sub>3</sub>O to  $\alpha$ - and  $\beta$ -thujaketonic acids or to the dicarbonic acids. The formation of aldehydes by the action of Ag<sub>3</sub>O on EtOH in alkaline soln. is small. Citral may be removed by aniline phosphate.

H. C. FULLER.

Products Containing Sugar and Alcohol (FREYER). 12.

Determination of Tartaric Acid (WARCOLLIER). 12.

U. S., 999,852, Aug. 8. F. PAMPE, Halle-on-the-Saale, Ger. Mashing malt and other grains for the manufacture of alcohol, by grinding the grain to fine flour,

supplying the flour to the cooker in a fine long stream causing the formation of a cloud of the flour in the upper part of the cooker, passing a spray of H<sub>2</sub>O from the center of the cloud outward therethrough as the dust settles in the cooker and collecting the wet flour in a conical body below the point where it is moistened and projecting a stream of steam centrally from the lower apex through the mass to give it a uniform circulation.

U. S., 1,000,596, Aug. 15. KARL GILO, Gross-Lichterfelde, near Berlin, and ERNST GILG, Steglitz, near Berlin, Ger. Making a beverage containing carbon dioxide and but little alcohol, by mixing a soln. containing sugar with blossoms having nectaries, e. g., blossoms of the lime or elder or linden tree, introducing air, allowing the yeast present in the nectaries to grow, interrupting the access of air and allowing the mixt. to ferment under pressure.

### 17. PHARMACEUTICAL CHEMISTRY.

#### VIRGIL COBLENTZ.

Commercial Digitalis Powder and its Preservation. A. JOANIN. Bull. sci. pharmacolog., 17, 707–17.—In testing the strength of digitalis, J. uses the Focke method by detg. the toxicity on frogs. Since the activity of the dried leaf is subject to great variations because of imperfect drying and preservation, the leaves should be dried so as to contain not more than 2% of  $H_2O$  and then pulverized and kept protected against moisture. The detn. of digitoxin does not suffice to show the medicinal value. Only powdered leaves of uniform strength should be marketed.

V. K. Chesnut.

Study on the Hydrocyanic Acid-Benzaldehyde Solution in Cherry Laurel Water. P. H. Worth. Arch. Pharm., 249, 382-407.—The equil. reaction of C<sub>6</sub>H<sub>5</sub>COHHCN in aq. soln. was verified. At the same conc. and temp. the same equil. was obtained. The dissociation of the cyanohydrin was greatest on dilution, the compd. being reformed on conc. The dissociation was accompanied by a rise in temp. The equil. reaction was (1) influenced by small amts. of alkalies and acids, the former increasing, the latter retarding; (2) most readily obtained by the action of alkali in the direction of hydrolysis of the cyanohydrin; however, it does not follow that a high conc. of alkali will cause a complete dissolution. AgNO<sub>5</sub> ppts. the available HCN out of alkaline soln.; after the free HCN is pptd. the dissociation of the cyanohydrin continues. In the prep. of cherry laurel and bitter almond water by distillation, benzaldehydecyanohydrin does not come from the glucoside as such, but in a hydrolyzed condition and the distillate contains at first HCN and C<sub>6</sub>H<sub>6</sub>COH. The rapidity with which the equil. is attained is in inverse ratio to the acidity of the distillate.

H. C. FULLER.

The Alkaloids of Pareira Root. M. SCHOLTZ. Arch. Pharm., 249, 408–18.—An acid soln. of the root was pptd. by NaOH and the mass extracted with Et<sub>2</sub>O and CHCl<sub>2</sub> to remove berberine. The residue after extraction with H<sub>2</sub>O was dissolved in dil. HCl and on pptg. with NaOH yielded chondrodin,  $C_{18}H_{10}O_4N$ , amorphous, m. 218–220°; almost insol. in Et<sub>2</sub>O, CHCl<sub>3</sub>, Me<sub>2</sub>CO, slightly in EtOH, readily in aniline and in NaOH,  $[\alpha]$ —75°. It gave no characteristic color reactions. It differs from berberine by the substitution of an H by OH. A number of salts and derivs were described. Ethylberberine and its iodomethylate were prepared.

H. C. F.

Rheum palmatum the Source of Good Officinal Rhubarb. C. C. Hosseus. Arch. Pharm., 249, 415-24. H. C. Fuller.

The Physical Constants of Bromoform of the German Pharmacopeia V. K. Feist

AND C. GARNER. Arch. Pharm., 249, 458-63.—CHBr, of the Ger. P. V. contains 4% EtOH and has d. 2.829-2.833, b. 148-150°, solidification point 5-6°. Expts. with CHBr, carefully purified and to which was added 4% EtOH showed d. 2.6354, b. 146.25°, solidification point 4°.

H. C. F.

Methyl Guanidines. M. SCHENCK. Arch. Pharm., 249, 463-80.—In attempting to prepare and isolate 3-methylguanidine only 1-methylguanidine resulted but it could only have been formed by a transformation of the first named during the reaction. Similarly, 1,2-dimethylguanidine resulted in an attempt to prepare 1,3-dimethylguanidine though the latter must have been formed in the reaction.

H. C. F.

The Composition of Blaud's Pill. A. E. PARKES AND J. D. ROBERTS. *Pharm.* J., 87, 320.—The exam. of a number of Blaud's pills showed that the official mass had been substituted by FeSO<sub>4</sub>.

H. C. F.

The "Pearl Coating" of Pills. A. E. PARKES AND J. D. ROBERTS. Pharm. J., 87, 320.—Magnesium silicate is used for the pearl coating of pills. H. C. F.

Notes on Indian Drugs. D. Hooper. Pharm. J., 87, 350.—The roots of Potentilla fulgans called "lyn-mang" are used as an astringent and contain 9.2% tannin. The seeds of Rumex maritima called "bijband" are used as an alterative and aphrodisiac. The fruit of Vanqueria spinosa is eaten for medicinal purposes, and contains sugar, gum and a trace of tannin. The resin of Podophyllum emodi runs 11.7-13.9%.

H. C. F.

Testing of Digitalis on Frog Hearts with Special Reference to the Varying Resistance of Temporariae towards Digitalis. FOCKE. Düsseldorf. Z. exp. Path., 9, 97-102; through Chem Zentr., 1911, 1I, 41.—The work contains recommendations for the improvement of Straub's method (C. A., 5, 1791) for testing digitalis on isolated frog hearts, and remarks on the author's method (Arch. der Pharm., 248, 365).

WALTER A. JACOBS.

Physiological Testing of Digitalis Preparations by Focke's Method. FOCKE. Schweiz. Wochschr., 49, 325-7; cf. C. A., 5, 3320 and preceding abstr.—Burmann's criticisms were based on the method as it was in 1903 and not as revized in 1910. The dose must be regulated so that heart action will cease in 7-14 mins. while Burmann's conclusions are based on expts. consuming 30-35 mins. The fact that when he treated frogs with a soln. of digitoxin containing 1 mg. and was able to recover from the lymph sacs 2-3 mg. unchanged showed that his doses were too large.

H. C. F.

The Physiological Testing of Galenical Preparations of Digitalis by Focke's Method.

J. Burmann. Schweiz. Wochschr., 49, 416-8.—1.5 cm. of dialyze Golaz (digitalis? strength?) injected into a frog produced heart cessation in 20 min., 2 cm. required 19 min., showing the relatively large doses required considerable time. The figures obtained by Focke's equation do not give the value of a cardiac medicin but express only the rapidity of absorption of the frogs employed.

H. C. F.

Nitras Bismuthicus Basicus. G. ROMIJN. *Pharm. Weekblad*, 48, 694-5.—The basic salt of the Holl. Pharm. may be distinguished from the other, by stirring with little H<sub>2</sub>O for 5 min. and then testing with Congo red paper. Basic preps. give no reaction.

V. E. HENDERSON.

Indole in Flower Perfume. J. SACK. Paramaribo. Pharm. Weekblad, 48, 775.—Indole is found in perfume of Hevea brasiliensis Muell and Randia formosa (Jacq.) K. Schum.

V. E. H.

Reactions for the Identification and Purity Estimations of Ferrum Reduction.

M. M. A. Cocx. Amsterdam. Pharm. Weshblad, 48, 776-9.—Criticism of the method

prescribed by the Pharmacopeia and also of those advocated by Schoorl (*Ibid.*, 1906, 1269) for the detection of pulverized Fe when mixed with or substituted for reduced Fe. Under the microscope, pulverized Fe gives a metal glance. The quant. methods are not accurate; that of Pharm. Germ. V is probably the best.

V. E. H.

The Quantitative Estimation of the Cinnamein Content of Balsam of Peru. D. VAN OS. Utrecht. *Pharm. Weskblad*, 48, 905-7.—A quant. estn. by various methods showing that that of the Holl. Pharm. is as good as the others. V. E. H.

Compressed Tablets. J. J. Hofman. Pharm: Weekblad, 48, 968-72.—Discussion of methods and hints on their prep. for the pharmacist.

V. E. H.

The Culture of Caraway Fruit in Holland. P. VAN DER WIELEN. Pharm. Week-blad, 48, 988-96.—An address describing the culture, amt. produced, and chem. constitution as compared with that grown elsewhere.

V. E. H.

Estimation of Iron with Iodine. G. ROMIJN. Bosch. Pharm. Weekblad, 48, 996-1000.—As it was found that Fe compds. in presence of organic materials could not be estd. by oxidation with KMnO<sub>4</sub>, the following method was devized: dissolve the sample in not more than enough H<sub>2</sub>SO<sub>4</sub> or HCl than would produce 20 cc. of N acid; to this add 20 cc. o. 1 N I and 5 g. powdered Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub> and shake in soln.; allow to stand 5 min. and then titrate the excess I with 0.1 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. The Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub> greatly hastens the rate of reaction. The results were approx. 0.2% too small. The quantity of acid and pyrophosphate must vary concordantly. Greater excess of I does not improve the result.

V. E. H.

Reactions for Indole. H. L. VISSER. Nijmegen. Pharm. Weekblad, 48, 1000-3.

—Various reactions are discussed and attention called to the fact that the Jacquemin-Dragendorff reaction in which hypochlorite soln. and then ammoniacal phenyl soln. are added, may be varied to act as a very delicate test for NH<sub>2</sub> as follows: Add hypochlorite to soln. containing NH<sub>4</sub>Cl; then NH<sub>2</sub> gives a distinct blue. The more dil. the NH<sub>2</sub> conc. the more dil. the hypochlorite must be.

V. E. H.

The Preparation of Extractum Belladonna. H. J. M. BARTELS AND P. VAN DER WIELEN. Pharm. Weekblad, 48, 1018-21. V. E. H.

The Water-absorbing Powers of Salve Constituents. H. J. M. Bartels and P. van Der Wielen. Pharm. Weekblad, 48, 1021-5.—White vaselin, as chief constituent, was melted together with 2.5-5% of beeswax, beeswax alc., cetaceum, cetaceum alc., adeps lanae or its alcs. Vaselin +2.5% wax absorbed 38% H<sub>2</sub>O (+5%, 42%; +10%, 35%); +2.5% melissylic alc. 75% (+5%, 80%; +10%, 65%); +2.5% cetaceum (about the same as vaselin alone), 30% (+5%, 40%; +10%, 35%); +2.5% acetyl ale. 80-88% (+5%, 80%; +10%, varied with slowness of adding H<sub>2</sub>O from 76 to 108%); +2.5% adeps lanae, dependent on rate of addition 80-140% (+5%, similarly, 100-224%; +10%, 140-288%). Alcs. prepared from adeps lanae were less efficient  $\pm 135\%$ . Addition of phenols, especially resorcinol, decreases greatly the H<sub>2</sub>O absorbability.

The Investigation of Oleum Terebinthinae. P. VAN DER WIELEN. Pharm. Week-blad, 48, 1026-31.—Criticism of the methods proposed in various pharmacopeias for detg. the purity of turpentine oil.

V. E. H.

Digest of Comments on the Pharmacopeia of the United States of America and the National Formulary for 1908. M. G. MOTTER AND M. I. WILBERT. Hyg. Lab., Pub. Health and M.-H. S. Bull. 75, 564 pages.

J. J. M.

Cera alba (White Wax) and Cera flava (Yellow Wax) of the German Pharmacopela,

EGIO. G. BUCHNER. Munich. Z. Offent. Chem., 17, 311.—It is stated that yellow beeswax must have an acid no. of 18.7-24.3, and an ester no. of 72.9-76.7 with a ratio of 3.6-3.8. White wax must have values of 18.7-22.4 and 74.8-76.7, also with a ratio of 3.6-3.8. The above values give for yellow wax a ratio of 3.9-3.15 and for white of 4.0-3.42. The conditions cannot therefore be complied with.

D. S. PRATT.

Dyeing Hair (BELTZER). 25.

Gum and Gelatin (TIEBACK). 28.

Testing Thujone in Absinthe (PHILLIPE, FALLENBERG). 16.

Estimation of Quinine (COCKBURN, BLACK). 11.

Destruction of Iodine (FOURTON). 6.

Products-Containing Sugar and Alcohol (FREYER). 12.

Lactore (BURR, BERBERICH). 12.

- U. S., 999,955, Aug. 8. J. CALLSEN, Elberfeld, Ger. Assignor to Farben. vorm. F. Bayer & Co., same place. Glyceryl monocinnamate, of the formula C<sub>2</sub>H<sub>2</sub>.CH.CH.-COO.CH<sub>2</sub>.CHOH.CH<sub>2</sub>.OH, a yellowish oil sol. in alc., ether and CHCl<sub>2</sub>, used as a remedy for scabies; made by heating cinnamic acid 148 with glycerol 400 and conc. H<sub>2</sub>SO<sub>4</sub> 20 parts to 140° for 60 hrs., sepg. and distilling in vacuo.
- U. S., 1,000,040, Aug. 8 P. Schickler and H. Hahl, Elberfeld, Ger. Assignors to Farben. vorm. F. Bayer & Co., same place. Glycol cinnamate, C<sub>6</sub>H<sub>6</sub>.CH.CH.COO.-CH<sub>7</sub>.CH<sub>8</sub>.OH which is a colorless and odorless oil b<sub>11</sub> 190-195°, used as a remedy for scabies; made by heating glycol 500 with cinnamic acid 250 and conc. H<sub>2</sub>SO<sub>4</sub> 190 parts to 140° for 36 hrs., sepg. and distilling in vacuo.
- U. S., 1,000,214, Aug. 8. C. E. VANDERKLEED, Collingswood, N. J. Assignor to H. K. Mulford Co., Philadelphia, Pa. Obtaining a blood-pressure-raising and hemostatic solution from suprarenal glands by acting on the ppt. obtained by treating an aq. ZnSO<sub>4</sub> ext. of the suprarenal gland with NH<sub>2</sub>, with an aq. soln. of H<sub>2</sub>BO<sub>3</sub> and filtering out the Zn borate formed.
- U. S., 1,000,423, Aug. 15. O. B. MAY, New York, N. Y Forming a solid oil-containing medicin by dissolving 33 parts of modified starch in  $H_2O$ , adding gluten 33 and oil, e. g., cod liver oil or castor oil, 33 parts, and drying the product.
- U. S., 1,000,673, Aug. 15. A. W. DIACK, Detroit, Mich. Pyrometer, comprizing a sealed glass tube containing a small body of phenacetin, acetanilide or S and a dye; intended for use in rolls of bandages, etc., which are to be sterilized by heat.
- U. S., 1,000,852, Aug. 15. A. STAPLER. Solid perfume mixture; see C. A., 5, 1825.

# 18. ACIDS, ALKALIES, SALTS AND SUNDRIES.

### T. LYNTON BRIGGS.

Forty-seventh Annual Report on Alkali, etc., Works by the Chief Inspector. Proceedings during the Year 1910. J. Soc. Chem. Ind., 30, 890-1. J. J. M.

Some Problems in Chemical Engineering Practice. F. W. FRERICHS. J. Ind. Eng. Chem., 3, 774-83.

J. J. M.

Catalytic Preparation of Ammonia from Compounds of Ritrogen and Carbon ee

of Ritrogen, Carbon and Hydrogen. F. Schreiber. Chem. Ztg., 35, 943.—Upon making compds. containing N with a large excess of hydrated oxide of Fe and heating in a wrought Fe tube to 350°, 46-73.7% of the N present was converted to NH<sub>2</sub> which could be recovered by aspirating air through the tube. The compds. used were pyridinosulfinic acid, albumin, K<sub>4</sub>Fe(CN)<sub>4</sub> and Fe<sub>7</sub>(CN)<sub>18</sub>. By conducting coke oven gas through the charged tube heated to 350°, the N compds. contained therein were converted to NH<sub>2</sub>.

A. F. Serker.

The Cheshire Salt Industry. Anon. Eng. Mining J., 92, 633-4.—Description of salt making from the brine streams of Cheshire. To be profitable for salt making the brine must yield at least 2.5 lb. of salt per gal.

ROBERT KANN.

Balloon Gas. W. P. JORISSEN. Chem. Weekblad, 8, 625.—The article is an excellent review of the methods, patents, etc., of making H on a com. scale.

E. C. L.

Studge Acids (PILAT, STARKEL). 22. Stirring Apparatus (LEISER). 1.

- U. S., 1,000,295, Aug. 8. W. RUMEL, Salt Lake City, Utah. Composition for insulating boilers, steam pipes, etc., consisting of "lime putty" 65%, sawdust 30% and plastering hair 5%.
- U. S., 1,000,298, Aug. 8. L. SARASON, Hirschgarten, near Berlin, Ger. Composition for slowly liberating oxygen, consisting of cryst.  $Na_4P_9O_7$  and  $Na_2B_4O_8$  + 10  $H_9O$  or other compd. containing loosely combined O.
- U. S., 1,000,598, Aug. 15. J. B. Hall, Philadelphia, Pa. Making a fibrous composition for use as a non-conductor of heat and electricity, by forming a milky mixt, of clay and H<sub>2</sub>O, emulsifying oleaginous or resinous material, e. g., hydrocarbon oil, with the mixt., adding a viscous substance, e. g., glue, and finely comminuted fibrous material, e. g., paper, leaves, peat or bark, and finally expelling the H<sub>2</sub>O.
- U. S., 1,000,732, Aug. 15. F. HÄUSSER, Kaiserslautern, Ger.; cf. C. A., 5, 2706, 2709. Making nitric acid by compressing a mixt. of N, O and a combustible gas, igniting the mixt., allowing the temp. to rize to the point necessary for producing nitric oxide and maintaining it at this point (1900–2000° or higher) until the nitric oxide is produced, suddenly causing the temp. to drop to a point below that at which nitric oxide is decomposed, conveying the nitric oxide into dil. HNO, and concentrating the latter by the heat of the exhaust gases.

## 19. GLASS AND CERAMICS.

### G. E. BARTON, A. V. BLEININGER.

The Machanical Production of Window Glass. B. SCHNEIDER. Glasind., 22, Nos. 32-5 (3 illus.).—The Sievert process is explained at some length and the Fourcault and American machines are compared to it. The cylinder of glass formed in the Sievert machine is subject to 3 defects: Unequal distribution of the glass, bubbles entering as the glass is poured and a dimmed surface from contact with the steel table of the machine. A note attached to the article contains a claim from the Sievert Co. that the 3 defects in their glass mentioned above have been removed.

J. B. PATCH.

The Fire Polishing of Sand Blast Designs upon Sheet Glass. Anon. Diamont, 33, 764-5.—By means of fire polishing a satin finish is obtained superior to that of ordinary mat glass. A machine is proposed and explained for applying the pattern to the outside of window glass cylinders which are later placed in the flattening oven where the fire polishing takes place, thus saving an operation.

J. B. P.

The "Schoop-Process" with Reference to the Production of Metallized Sheet Glass and Stone Articles. F. HERKENRATH. Diamant, 33, 799-800.—The writer points out the special application of the process in the manufacture of mirrors and unique tombstones; cf. C. A., 5, 3138.

J. B. P.

The Treatment of the Mussle. CARL FALK. Glassed., 22, No. 35. J. B. P.

Majolica Glazes upon Cast Iron. PH. EYER. Glashatte, 41, 737-8, 764-5.-E. first describes in detail the pickling and washing of the cast Fe article. The foundation layer must be put on with care to protect the Fe from the action of the constituent of the white glaze, as for example the reduction of Sn oxide by the C of the Fe resulting in bubbles in the enamel. A recipe is given which does away with the 2 layers and sep. firing. It contains quartz 17.7, feldspar 18.6, borax 35.0, Na silicofluoride 2.5, KNO, 0.9, Ni oxide 0.3, Co oxide 0.1 and Cu oxide 0.002. In mixing, 7% clay is added. It is believed that the Co and Ni form a weak alloy with the Fe and protect it from the action of the white glaze. White glazes containing Sn oxide may be made more fusible by the addition of cryolite or feldspar so that the temps, of firing may be lowered and the formation of bubbles as explained above prevented. Another method of avoiding the 2 sep. firings is to add water glass to the first layer so that after simple drying of the foundation glaze the white glaze may be added and fired. The majolica glaze is placed upon white glaze obtained by any of the above methods and its comp. must be carefully regulated to avoid crazing and shivering. There is danger of adding an excess of H<sub>2</sub>BO<sub>2</sub> to a majolica glaze containing Pb which causes the resulting glaze to be attacked by moisture which removes Pb borate. Borax is preferably used in this case although the luster produced is not so brilliant. A number of recipes are given. J. B. P.

- U. S., 1,000,378, Aug. 15. G. C. DE BAY, Tarentum, Pa. Assignor one-half to J. Heidenkamp, Springdale, Pa. Device for polishing hot plate glass, formed of fine vegetable charcoal and plaster of Paris, molded into a firm body with holes for C loosened in the polishing to pass through.
  - U. S., 1,000,546, Aug. 15. P. J. PAQUET, Jeannette, Pa. Glass furnace.
- U. S., 1,000,671, Aug. 15. M. W. DAVISON, G. KESTER and J. M. ROWAN, Dubois, Pa. Device for tempering glass.
- U. S., 1,000,921, Aug. 15. W. W. W. KEYES, Alexandria, Ind. Glass melting apparatus.

### 20. CEMENT AND OTHER BUILDING MATERIALS.

### C. N. WILEY.

Physical and Chemical Properties of Portland Cement. III. W. C. REIBLING AND F. D. REYES. *Philipp J. Sci.*, 6 (A), 207-50.—The authors have made an exhaustive study of the causes which affect the setting of Portland cement and discuss at length the influence which various proportions of CaO, Ca(OH), and CaCO, in the cement have on the variation in setting time. They assert that a great deal of cement

which develops quick setting when tested on the work has escaped the attention of the manufr. by being unconsciously regaged when mixed for testing at the factory lab. Many manufrs. add too much gypsum to the cement, which latter often later becomes quick-setting due to having an excess of gypsum. They advance a new theory as to the influence of gypsum and in discussing the theories of Rohland and Candlot say "that it would seem as if the action of gypsum was capable of a simpler explanation than those already advanced. The salt is sol. in water to the extent of 1 part CaSO<sub>4</sub> to 490 parts water at 15°. In dissolving it is ionized and obviously the addition of Ca ions would reduce the solubility of the other Ca ions already present. The free Ca would therefore be less rapidly dissolved and the hydrolysis of the aluminates would be retarded, the result being a slowing of the set. As the free Ca changes to CaCO<sub>3</sub> it is removed from soln. and becomes inert. The loss of these Ca ions, of course, increases the solubility of the aluminates and the facility with which they hydrolyze, the result being the quickening of the set."

C. N. Wiley.

Cement Boiling Test According to Michaelis. H. KÜHL. Chem. Zig., 35, 815-6.

—All accelerated tests of cement being empirical in character, the author considers all such modifications as that proposed by Hentschel (C. A., 5, 2540) as uselessly complicating the already well established Michaelis test.

A. J. PHILLIPS.

Chemistry of Iron Concrete. E. Donath. Z. angew. Chem., 24, 1398-1402.-The analyses of 4 rust samples show resp.: 74.52, 86.45, 78.56 and 92.94% Fe<sub>2</sub>O<sub>2</sub>, the remainder being -H<sub>2</sub>O, +H<sub>2</sub>O, CO<sub>2</sub>, SiO<sub>2</sub>, P<sub>2</sub>O<sub>3</sub>, SO<sub>3</sub>, FeO, Mn<sub>2</sub>O<sub>3</sub>, graphite and NH<sub>2</sub>. The presence of acid constituents and of FeO is emphasized with regard to later conditions. Rohland's work (C. A., 4, 881; 5, 583, 1177) is reviewed. R. neglects the probable reduction of at least a part of the Fe to the ferrous state. Several samples of rust were treated with (1) a Ca bicarbonate soln. containing excess of CO, (2) the filtrate from 200 g. of cement shaken with 2 l. H<sub>2</sub>O. A portion of the soln, which gave reddish surface films due to oxidation, on acidulation with HCl gave the ferrous Fe reaction with K.FeCN, showing that FeO not Fe,O, first goes into soln. The half bound CO<sub>2</sub>(HCO<sub>2</sub>) dissolves the rust present as FeO while the Ca dissolves the acid constituents and loosens the firmly compacted scales. Dunkelburg's NH, absorption theory is reviewed (C. A., 5, 1329). In case NH, is absorbed, the bacteria in the rust may oxidize it to HNO, and HNO. To test the theory of Michaelis (C. A., 3, 3003), 3 g. of rust and 6 g. CaO were mixed with water; after 4 hrs., 300 cc. HOAc were added and at same time a rust sample was treated solely with HOAc. Detn. of Fe in the filtrates showed 1.56% Fe<sub>2</sub>O<sub>3</sub> for rust alone and 2.46% for rust + CaO. Similar treatment, using 10 g. of cement instead of CaO, showed 3.70% Fe<sub>2</sub>O<sub>2</sub> and an alkaline sugar soln. gave 4.57% while a preliminary treat. with cement showed 6.54%. The conclusion is that from the mixt. of Fe oxides a hydroferrite is formed, the Ca decomposing and exposing fresh surfaces of FeO. A part of the Fe,O,-that part which the alkaline sugar dissolves—is also dissolved by the Ca. A. J. PHILLIPS.

Washable Plaster Casts. F. RATHGEN. Rep. 13th Gen. Meeting Gipsuer., 1911, 50-4; through J. Soc. Chem. Ind., 30, 622.—Washing plaster casts with soap and water spoils the contours, at least after 2 washings. R. prefers leaving the casts stand in water sadt. with plaster and then taking off the outer scum. Casts may be rendered washable by satg. the surface with wax or paraffin or some chem. which renders it less permeable. Treatment of the cast with "zapon" (a soln. of nitrocellulose) yields the best results, and when this is done it may be regularly washed with soap and water and a soft brush, but the treatment should be repeated every year or two as the surface cracks. Instead of "zapon," "acetylcellulose" ("cellon," "cellite") may be used. Smoke stains may be removed by alc. or petroleum spirit. C. N. Wiley.

The Use of Hydrated Lime in Portland Cement Mixtures. R. J. Wig. Tross. 9th Ann. Meet. Nat. Lime Mfrs. Assoc., 213-9.—The addition of up to 15%, based on the wt. of cement in the mortar, has the following effects: Tensil strength, not affected in lean mortars and decreased in rich mortars; compressive strength, considerably increased in both poor and rich mortars; adhesive strength, greatly increased in lean mortars by use of CaO; permeability reduced by the addition of lime more than by an equivalent wt. of cement; absorption of green mortars slightly reduced and absorption in aged mortars slightly increased; plasticity greatly increased. A. J. Phillips.

Tests of Lime. W. E. EMLEY. Trans. 9th Ann. Meet. Not. Assoc. Lime Migrs., 192-208.—As the result of the phys. and chem. testing of 38 samples of lime the following conclusions are drawn: (1) 2-in. cubes exposed to air on 5 sides were not fully carbonated at the end of 6 mo., (2) the d. of cubes of dolomite lime is greater than that of high Ca limes due either to large % of mixing water in the latter or to the greater d. of MgO. (3) The shrinkage factor is lower with dolomite limes than with high Ca limes. (4) The crushing strength varies directly as the d. (5) Mg limes are stronger than high Ca limes.

A. J. P.

The Burning Temperature of Limestone. A. V. BLEININGER AND W. E. EMLEY. Trans. 9th Ann. Meet. Nat. Assoc. Lime Mfrs., 68-81.—(1) The min. calcination temp. of high Ca stone was found to be 880° and of MgCO<sub>3</sub> 750°. (2) The limiting burning temps. of limes may be detd. from a study of the hydration curves of lime burnt at diff. temps. In these the temp. rise of a certain wt. of water heated by slaking a given amt. of lime is plotted against time; thus giving a method of fixing the proper burning temp. of a given lime. (3) The max. temp. permissible for high Ca stones is above 1260°, for impure stones 1040-1200°, for pure Mg stone 1000-1200° and for impure Mg stone 900-1200°. (4) The point of max. porosity on the porosity-temp. curve of a lime indicates the best burning temp. The steeper the slope of this curve the greater the tendency toward overburning and vice versa. (5) The d. curve of a limestone confirms the evidence under (4) and gives additional information as regards the burning of Mg limestones. (6) The more porous a limestone is the more rapidly it will burn and at a lower temp. Rate of hydration, porosity and sp. gr. curves are plottad.

A. J. P. I. I. M.

A Pole-preserving Machine. Anon. Eng. News, 66, 414-5.

U. S., 1,000,386, Aug. 15. R. EGGENHOFFNER, Genoa, Italy. Magnesia coment containing Mg<sub>2</sub>OCl<sub>2</sub>.H<sub>2</sub>O and MgO, preferably in the mol. proportions of 1 and 7 resp.

U. S., 1,000,425, Aug. 15. B. MELZER, Hetschburg, near Weimar, Ger. Making solid colored bodies in imitation of wood, stone, etc., by squirting onto an elastic foundation, e. g., wire or rubber, superposed layers of comps. of different colors, applied from different directions, and finally polishing the surface of the mass so produced, after drying. Colored mixts. of paper pulp and casein may be employed.

U. S., 1,000,520, Aug. 15. G. P. Hemstreet, Hastings-upon-Hudson, N. Y. Assignor to Intern. Pavement Co., Hartford, Conn. Making a paving composition by heating a body material of stony matter and fines, dry mixing and adding a cementitious binder, compressing into blocks or tiles, cooling them and permitting them to set, breaking them into pieces 1 to 2 in. in thickness, heating the pieces together uniformly, mixing with a fresh batch of the original heated uncompressed comp., and compressing the resulting mixt. for use.

U. S., 1,000,545 (dedicated to the public), Aug. 15. L. W. PAGE, Washington,

- D. C. Preparing hydraulic cement concrete by mixing cement, sand, aggregate and  $H_2O$  in the usual way and then adding to the mixt. before it begins to set a non-volatil mineral oil in quantity not more than 25% of the cement.
- U. S., 1,000,944, Aug. 15. J. M. RAUHOFF, Chicago, Ill. Assignor to Ironite Co., Chicago, Ill. Concrete composition comprizing a cementitious comp., fine Fe dust and asbestos.

## 21. FUELS, GAS AND COKE.

### J. D. PENNOCK.

Heating Value of Fuels. Rules of the French Coke Committee. Gas World, 55. 111-2.—(a) Starting with a fuel which dried at 110°, find the gross heating value in cal. per kg., the water from the combustion being supposed to be cooled to the f. p.; this is the abs. heating power of the dried fuel, or the abs. heating power, simply. Suppose this to be 6882 cal. per kg. (b) Let a fuel containing 12% of water be given, dry it at 110°; the abs. heating value of the dried residue is, again, 6882 cal. per kg. Then the heating value of the original wet fuel is only 6056 cal. per kg. This is termed the abs. heating power of the crude, damp fuel. (c) Let a fuel containing 12% of moisture, 13% of ash, and 75% of combustible matter be given, and let its abs. heating power (a above) be 6882, or its abs. heating power when damp (b, above) be 6056; then the heating power per kg. of the combustible material (excluding moisture and ash) is 8075 cal. per kg. Term this the abs. heating power of the pure combustible. This is of importance in reference to deterioration through exposure to moisture. If the moisture rizes to 22 instead of 12%, the heating power wet (b, above) falls from 6056 to 5368. (d) The lab. bomb test gives a figure which assumes that the water formed has cooled to o° and has given up all its heat. This is not the practical result of burning the coal as fuel; steam or H<sub>2</sub>O vapor carries off heat units. Each g. of steam at 100° carries off 0.637 cal., whether it owes its origin to the original moisture or to the combustion of H. On deducting this, we arrive, from the abs. heating power (a, above), at the net heating power—the useful heating power of the combustible. (e) Given a coal containing 12% of moisture, 13% of ash, and 75% of combustible matter, and assuming this latter contain 1% of H, then on burning 1 kg. of this there would be evapd. 0.12 kg. of H<sub>2</sub>O, and 0.0675 kg. of H<sub>2</sub>O vapor produced by the combustion; in all, 0.1875 kg.  $H_2O$  vapor at 0.637 cal. per g., equals 187.5  $\times$  0.637 cal., equals 119 cals., and the heating value of the wet fuel (b, above) is 5937 instead of 6056. This is the useful heating power of the crude damp material. This is the most useful of all the values. The H<sub>2</sub>O vapor produced by the combustion may cause the practical value of the fuel to be as much as 8% less than that given by Mahler's bomb (b, above). The thermal efficiency of any app. is the ratio between the number of cal. utilized and the number given by the fuel, in a given period of time. The latter number is the product of the wt. of the fuel into its heating value. This heating value may be the abs. (d, above), in which case we have the "abs. thermal efficiency." The coke Committee announces its intention of using, in all specifications, the useful heating power (e, above) in stating this ratio, which will then be called the useful thermal efficiency.

R. E. H.

Utilization of Canada's Peat Bogs. Anon. Elec. Rev. West. Elec., 59, 459.—The estimated peat bogs of Canada, cover approx. 36,000 sq. mi., and are capable of producing 28,000,000,000 tons of air-dried peat, equal in fuel value to 14,000,000,000 tons of coal. A description of a well equipped plant at Ottawa for producing air-dried peat is given.

W. L. VAN KEUREN.

Solid Fuels in 1910. Bertelsmann and Hörmann. Chem. Zig., 35, 1021-2.
J. J. M.

Smoke Prevention. H. M. NICKOLLS. Iron Age, 86, 1157-8.—Modifications (described and illustrated) in the setting of the boiler and heating furnace in an Fe and steel plant, resulted in the prevention of smoke in this case. L. A. TOUZALIN.

Gas Producer Design and Operation. P. von Zeipel. Iron Age, 86, 1154-5.

—Historical review.

L. A. T.

The Cedford Gas Process. E. ERDMANN. Lab. angew. Chem., Univ. Halle a/S. J. Gasbel., 54, 737-41; Gas World, 55, 195-7.—The object of the process is to produce CH<sub>4</sub> from CO and H on a com. scale. Water gas, carefully purified, dry and CO<sub>2</sub> free, is treated in a special Linde app. Here it is sepd. into a gas rich in CO and ene containing 17% CO and 81% H. This latter gas is passed over a catalytic mass consisting of pumice stone covered with Ni, and heated to 300°. When the temp. is below 300° and the CO content below 17%, the CO is changed quantitatively into CH<sub>4</sub>. When the temp. is too high, CO<sub>2</sub> is formed, and when the CO content is too great C is deposited. The CO-rich gas is used for fuel around the plant. The chief advantages are (1) the poisonous CO is removed, making the gas much safer, (2) the heat value is increased (from 2800 to 8500 cal.). Cost data are given.

GEORGE W. MOREY.

Gas Analysis by Fractional Combustion. L. UBBELOHDE AND DE CASTRO. Chem-Tech. Inst. Techn. Hochsch. Karlsruhe. J. Gasbel., 54, 810-4—The combustion is made in 2 parts, using CuO as catalyzer. H + CO are first burned at  $265-70^{\circ}$ , then the CuO is heated to  $800-900^{\circ}$  and  $CH_4 + C_4M_6$  detd. From the change in vol. followed by a CO<sub>2</sub> detn. the amts. of the gases are calc. The app. and methods for the complete analysis of illuminating gas are described in detail. George W. Morey.

Direct Recovery of Ammonia as Sulfate. Anon. Gas World 55, 76-7.—Description of the Burstall app. at Birmingham Univ. GEORGE W. MOREY.

Contamination of Gas in Holders by the Hydrogen Sulfide Evolved from the Water in the Tank by Bacterial Processes. J. G. Taplay. Gas World, 54, 105-7.—H<sub>2</sub>S is produced by the action of B. coli communis and B. proteus on MgSO<sub>4</sub> or CaSO<sub>4</sub> dissolved in gasholder tank waters, and the amt. produced may seriously contaminate the gas. The organism can be completely destroyed by addition of CuSO<sub>4</sub>, 1 part by wt. to 10,000 of H<sub>2</sub>O.

George W. Morey.

Naphthalene Washing. ZWARG. Elbing. J. Gasbel., 54, 837-8.—Expts. on the use of an oil gas tar as wash oil for naphthalene removal. The use of such a tar alone is not advizable since it leaves at least 4 g.  $C_{10}O_3$  per 100 cu. m. in the gas.

GEORGE W. MOREY.

Determination of Naphthalene in Crude Coal Gas. ARPAD WEIN. Städt. Gaswerke Budapest. J. Gasbel., 54, 891-2.—A modification of the picric acid method. The crude gas is drawn through picric acid and the naphthalene picrate obtained in the ordinary manner. It is filtered and transferred to an app. consisting of an Erlenmeyer flask, trap, and wash bottles filled with standard picric acid soln. To the naphthalene picrate in the Erlenmeyer flask is added H<sub>2</sub>O containing enough H<sub>2</sub>SO<sub>4</sub> to fix the NH<sub>2</sub>, the naphthalene is freed by b. and drawn into the wash flasks by a current of air. The ppt. is filtered off and excess picric acid detd. iodometrically by the method of Schlumberger.

George W. Morey.

New Gas Analysis Apparatus. I

Apparatus for Density and Molecular Weight of Gases (GULICH). 1.

- U. S., 999,784, Aug. 8. J. A. HERRICK, New York, N. Y. Gas producer and blast-box structure therefor.
- U. S., 999,807, Aug. 8. H. I. Lea, Pittsburgh, Pa. Gas producer having at the top of the gas generating chamber a steam generator formed with a spiral groove increasing in depth and cross-sectional area from its inlet port to its outlet port.
- U. S., 999,973, Aug. 8. J. P. FARMER, Portland, Ore. Assignor one-fourth each to P. J. G. Kleppin and Joseph Wick, same place. Gas purifier.
- U. S., 1,000,479, Aug. 15. A. ZINDLER, Berlin, Ger. Briquetting peat and non-briquetting material, such as anthracite coal or Fe ore, by mixing the materials, heating to a temp. sufficient to liberate bitumimous binding material from the peat, and forming into briquets.
- U. S., 1,000,655, Aug. 15. L. Wolf, Zurich, Switz. Assignor to Schweiz. Flussigas-Fabric L. Wolf A. G., same place. Making a liquefied gas from distillation gases by passing them through a cooling chamber and therein subjecting them to the cooling effect of expansion and vaporization of highly compressed hydrocarbon fluid, and then introducing the expanded and vaporized hydrocarbon into the distillation gases to be cooled before the latter are subjected to the cooling process.
- U. S., 1,000,669, Aug. 15. A. S. Cooper, Los Olivos, Cal. Apparatus for obtaining gas from wells.
- U. S., 1,000,768, Aug. 15. E. B. BENHAM, New London, Conn. Assignor to Hydrocarbon Converter Co., New York, N. Y. Making gas from hydrocarbon oils and other liquid carbonaceous materials, by maintaining incomplete combustion of the material to be gasified in a high temp. zone, supplying material for gas formation to the zone by passing an intimate mixt. of air and the material to be gasified (the latter in the form of minute drops) before the liquid has reached a high temp., in heating proximity to an extended highly heated surface of porous material and causing the resulting product to pass into the region of combustion, at a rate such that combustion begins beyond the immediate vicinity of the point of admission.

# 22. PETROLEUM, ASPHALT, COAL TAR AND WOOD PRODUCTS.

### R. E. HUMPHREYS.

The Removal of Water from Crude Petroleum. A. Beazley. Oil Age, 1911, 21; through Petroleum, 6, 2076.—B. describes the elec. process of Cottrell and Speed. After the water that seps. out from the oil by gravity is drawn off, the oil is pumped into vertical cylinders of special form, fitted inside and outside with specially constructed electrodes. A current of 10-15,000 v. is sent through the oil. Short circuits are prevented by rotating the electrodes. The minute drops of water, as conductors of the current, arrange themselves chain-like from electrode to electrode along the electrostatic lines, and form larger drops which sink to the bottom of the cylinders. The only change in the oil is the lowering of its sp. gr. All the salts, which formerly caused serious injury to the stills, are also removed along with the water.

O. E. BRANSKY.

Petroleum Trade in Germany in 1910. Anon. Chem. Tech. Zig., 29, 125.

O. E. B.

Specifications for the Purchase of Fuel Oil for the Government. I. C. ALLEN. Bur. of Mines. Techn. Paper No. 3.—Fuel oil should: (1) be either a natural homo-

geneous oil or a homogeneous residue from a natural oil; if the latter, all constituents having a low flash point should have been removed by distillation; it should not be composed of a light oil and a heavy residue mixed in such proportions as to give the d. desired; (2) not have been distilled at a temp, high enough to burn it, or to sep, flecks of carbonaceous matter; (3) not flash below 60°, using a closed cup; (4) have sp. gr. of 0.85-0.96 at 15°; (5) be free from solid or semi-solid substances, and should flow readily, at ordinary temps, and under a head of 1 ft. oil, through a 4-in. pipe 10 ft. long; (6) not congeal nor become too sluggish to flow at 0°; (7) have a calorific value of 10,000 cal. per g. (18,000 B. t. u. per lb.); 10,150 cal. to be the standard; (8) not contain more than 2% of H<sub>2</sub>O; (9) or 1% of S; (10) not contain any trace of sand, clay or dirt.

Chemical Composition of Mineral Oils of High Boiling Point. J. MARCUSSON. Chem. Ztq., 35, 729-31; through J. Soc. Chem. Ind., 30, 943.—The constituents to which mineral machine oils chiefly owe their lubricating power are those not reacting with CH<sub>2</sub>O and H<sub>2</sub>SO<sub>2</sub>. The cyclic unsatd. hydrocarbons reacting with these reagents to form sparingly sol. "formolites" have a higher d. and \* but are more fluid than the original oils. In applying the test to viscous oils, 27 g. of the sample are dissolved in 50 cc. of petroleum spirit and the soln, treated, without shaking, with 30 cc. conc. H<sub>2</sub>SO<sub>4</sub>; the mixture is then cooled in ice-water and treated, little by little, with constant shaking and cooling, with 15 cc. of 40% CH<sub>2</sub>O soln. After transferring to a 1. flask containing 200 cc. ice-water, the acid liquid is neutralized with NH<sub>4</sub>OH, and the ppt. collected, drained, washed free from NH,OH, and dried at 105°. to const. wt. Russian machine oils yielded 10-24% of "formolite," American oils 30-33%. As a rule, American oils are also richer in unsatd. hydrocarbons which combine with I. Thus, 11 samples of American oils gave I values of 6.1-15.3, as against I values of 3.3-5.6 in the case of 4 Russian oils. The I absorption is due not only to unsatd. alicyclic hydrocarbons, but also to aliphatic olefins. Russian oils contain the larger proportion of hydrocarbons that resist the action of fuming HNO<sub>a</sub>.

Transformer Oil. A. DUCKHAM. Electricion, 67, 212.—Mineral oils have been found best for use in transformers. The oil must be quite thin and mobil for the circulation must be rapid, but since it has to be worked at high temps., it must not give off appreciable vapor. For highest dielec. strength, all H<sub>2</sub>O, even that of satn., must be removed. This puts the oil in very unstable condition. Deposits or sludges and thickening of the oil are extremely objectionable and are caused by (1) deposit of atmospheric dust, (2) thickening due to evapn., (3) action of the oil on insulation and varnishes of the transformer, and (4) chem. changes in the oil. The use of sol. and insufficiently dried varnishes in insulation was found to cause a large amt. of saponifiable matter as Pb and Mn soap. These metal radicals came from driers used with the varnishes. Mineral oils become, under conditions of operation of a transformer, more or less oxidized and saponified. This is to some extent due to O, and NO,

W. E. RUDER.

Petroleum in Southeastern Russia. Anon. Petroleum Rev., 24, 326.—A spouter of tremendous power has been developed which indicates a petroliferous area of the R. E. H. greatest importance.

The Petroleum Industry of Baku and Grosny. Petroleum Rev., 24, 331-2. R. E. H.

The Russian Petroleum Industry during 1910. Petroleum Rev., 24, 342.—Statistics R. E. H. of production, refining and export.

The Tampico Oilfield, Mexico. S. H. BALL. Eng. Min. J., 91, 959-61; through R. E. H. J. Soc. Chem. Ind., 30, 733.

The Sludge Acids of Oil Refineries. St. von Pilat and W. Starkel. Petroleum, 6, 2177-9.—Wide differences are noted between the results obtained by titrating the sludge acids with alkali and those obtained by detg. the acid content gravimetrically as BaSO4. In the case of an acid used in the refining of benzine, total acidity by titration amtd. to 90.03%, gravimetrically to 78.6%. Variations from 1 to 11.43% were found. Since no aromatic sulfo acids could be isolated from the filtrate of BaSO. it is concluded that these sulfo acids are not formed under the conditions existing in the refineries. Egeleanu and Gane obtained a no. of aromatic sulfo acids from sludge acids, but in their expts. the oils were treated with fuming H,SO, at 80°. The authors think that these acid compds. are either addition products of H.SO, residues with unsatd. hydrocarbons or substitution products of H<sub>2</sub>SO<sub>4</sub> through organic radicals. No change was observed in samples of sludge acid after standing undisturbed for a month. Blowing with air only slightly affected the acids. The tarry upper layer that forms upon dilg. the sludge acid was found to contain organic sulfites by treatment with Pb(OAc). Besides these compds., the chief constituents are polymerized unsatd. hydrocarbons. They conclude that the H<sub>2</sub>SO<sub>4</sub> acts chiefly as a polymerizer.

O. E. BRANSKY.

Preparation of Oxalic Acid by Fusing Sawdust with Potassium Hydroxide. A. VON HEDENSTRÖM. Chem. Ztg., 35, 853-4.—A series of investigations to det. the conditions under which the best yields of H<sub>2</sub>C<sub>2</sub>O<sub>4</sub> are to be obtained by the fusion of cellulose materials with KOH. 3 g. of purified cotton were soaked in KOH soln. (12 g. KOH per 20 cc. water) and heated in a Ni crucible. The best yield (3.72 g. H<sub>2</sub>C<sub>2</sub>O<sub>4</sub> from 3 g. cotton) was obtained when the mass was heated slowly and with stirring to 280° and, after action had ceased, was cooled slowly. A white powder was thus obtained, sol. in H<sub>2</sub>O, the soln. giving no ppt. with dil. acids. The yield was not increased by the use of stronger KOH solns. and was greatly decreased when the fusion was carried on at 225°. When cotton was soaked in a soln. of 13 g. KOH and 1 g. H<sub>2</sub>C<sub>2</sub>O<sub>4</sub> in 30 cc. H<sub>2</sub>O and heated as before, the yield was considerably increased (5.16 g. H<sub>2</sub>C<sub>2</sub>O<sub>4</sub> from 3 g. Watte). The addition of such oxidizing agents as KMnO<sub>4</sub> and PbO, to the melt increased the yield 10-15%. The addition of KNO, or KClO, did not increase the yield. The conditions cited above applied also when oak sawdust was used as the raw material, the yield in this case being about the same as the yield from pure cellulose, vis., 3.66 g. H,C,O, from 3 g. sawdust. It appears therefore that the lignin constituents of the sawdust go also to form H<sub>2</sub>C<sub>2</sub>O<sub>4</sub>. This was further shown by allowing 3 g. sawdust to stand 3 days with 10% KOH soln., filtering, and evapg. the filtrate and wash H<sub>2</sub>O to dryness. A small yield of H<sub>2</sub>C<sub>2</sub>O<sub>4</sub> was obtained (1.81 g. H<sub>2</sub>C<sub>2</sub>O<sub>4</sub> from 3 g. sawdust). V. Nunez.

U. S., 1,000,646, Aug. 15. W. O. TH. VAN TIENEN, Rotterdam, Netherlands. Recovering hydrocarbons and sulfuric acid from acid tar (from mineral oil) by mixing therewith sufficient H<sub>2</sub>O to dil. the acid yielded by the tar to a sp. gr. of not more than 52° Bé., and heating the mixt. at a pressure of about 7 atm. and a temp. of about 140–165°. The liquid seps. into 2 layers, one containing regenerated oils and the other H<sub>2</sub>SO<sub>4</sub>.

## 23. CELLULOSE AND PAPER.

#### A. D. LITTLE.

Extracts from the Literature of Cellulose and Paper Chemistry. C. G. SCHWALBE.

Darmstadt. Papier Zig., 36, 2599-2601, 2631-2.

V. NUNEZ.

Manufacture of Celluloid. J. SCHMERBER. Caoutchouc and gutta-percha, 8, 4783-4, 4861-2, 4951-2, 5158-62, 5450-4, 5536-8. J. J. M.

Paper Specialties. H. Postt. Nuremburg. Papier Fabrikani, 9, 853-6.—Genuin Japanese paper is made by very gentle soda treatment of new fibers, such as the best fibers of the mulberry tree and others. Rags are not used, and Cl bleach liquor is not employed. A strong, smooth paper results. Dutch tobacco paper is made from cotton, linen and old paper stock. The paper is cut when moist, sized with animal size and air-dried. Specifications for this paper demand that it be strong and resistant to folding, have a light and permanent color and be non-porous. Imitation hand-made cardboard is required to weigh 350 g. per sq. m. Deckle edges on 4 sides and a definit grain are asked for. The paper is rosin sized. Parchment paper is also described.

Purification of Water (ACKERT). 14.

New Solvents for Cellulose (DEMING). 10.

- U. S., 999,986, Aug. 8. F. GIORDANO, Genoa, Italy. Making tobacco paper from tobacco stems by reducing them to a paste, removing resinous and incrustating matter as far as possible by mechanical means, eliminating any remaining incrustating substances by treatment with a 1% soln. of NaOH at 70-80°, draining off the soln., washing the paste, pouring a soln. of Na<sub>2</sub>SO<sub>2</sub> and SO<sub>2</sub> on it, heating it slightly and allowing it to stand some time, washing, refining and forming into paper.
- U. S., 1,000,628, Aug. 15. G. D. RYTHER, Carthage, N. Y. Paper-stock refining apparatus.
- U. S., 1,000,827, Aug. 15. C. R. LINEMBYER, Bremen, Ger. Making mixtures containing cupric hydroxide readily soluble in ammonia (and suitable for dissolving cellulose), by dissoving CuSO<sub>4</sub> in H<sub>4</sub>O, adding dil. NaOH soln. and then adding an aq. soln. of NaHCO<sub>2</sub> and subsequently additional dil. NaOH soln.

## 24. EXPLOSIVES.

C. E. MUNROE.

A Primer on Explosives for Coal Miners. CHARLES E. MUNROE AND CLARENCE HALL. U. S. Bur. Mines, Bull. 17 (1911).—Chemists who may wish to cite this in court practice should note that while the title page declares this a "Reprint of U. S. Geol. Sur., Bull. 423" it is revized in many particulars and that the very important "table of distances" is wholly new.

CHARLES E. MUNROE.

Detonating Explosives. LOTHAR WÖHLER. Darmstadt. Z. ges. Schiess-Spreag-stoffw., 6, 253.—Discusses particularly the action of detonating materials. The efficiency of such explosives depends on their own initial velocity of explosion. Thus the metallic nitrides, especially those of Hg, Ag, Pb, and Cu are far superior to Hg fulminate as initiating explosives, because of the greater rapidity of their detonation. Hg fulminate when very finely divided is threefold more efficient than when in its ordinary condition. Pb nitride has many advantages over Hg fulminate, as to sensitiveness, hygroscopicity, etc. The sensitiveness of the nitrides increases with their cryst. size. The action of light on nitrides of the heavy metals appears to be analogous to that of the Ag halides, finely divided metal resulting, and in the case of Hg nitride, metallic drops were obtained and photographed with the aid of the microscope.

C. G. STORM.

French Brisant Explosives. Hans Hessen. Z. ges. Schiess-Sprengstoffw., 6, 296-8.—The French Navy uses compressed guncotton as charges for torpedoes, mines, etc. Its properties, prep. and method of testing for brisanz on Fe plates are briefly described. The Army uses "melinite" and "cresylite" as shell explosives, fuse comps., etc., consisting chiefly of picric acid and trinitrocresol, resp. The prep., properties, etc., are discussed at some length. There are 3 types of "cresylite" and 2 of "melinite." All are used in the fused condition for loading. The common metals excepting Sn are more or less readily attacked, and in this fact lies the chief danger from the use of these explosives. The introduction of even small particles of metals, lime, nitrates, chlorates, etc., into a quantity of burning picric acid will cause a violent detonation which is transmitted to the entire mass. This fact may explain many explosions of picric acid during fires in factories and magazines.

C. G. Storm.

The Testing of Explosives. Bruno Zschokke. Z. ges. Schiess-Sprengstoffw., 6, 241.—Z. notes that in spite of recent great advances in the science of explosives, methods are lacking for satisfactory scientific measurement of important properties of explosives, e. g., sensitiveness to friction or blows, the detg. of explosive energy, the calc. of the necessary charge of explosive in different materials, etc. The need of cooperation among investigators is pointed out.

C. G. Storm.

The New Tests for Permitted Explosives. Arms and Explosives, 19, 115-6.—A demand for greater severity in the requirements of safety explosives for the English, "permitted list" has resulted in a series of expts. at the Woolwich testing gallery for detg. the best manner of modifying the gallery tests so as to insure greater safety for the miner. The new requirements will probably include the firing of stemmed shots separately into a gas mixt. and into a coal dust atm., and that a limit charge will be detd, for governing the max. amt. of each particular explosive that may be fired in a single bore hole. The factors influencing gallery tests in general are briefly discussed.

C. G. Storm.

Trinitrotoluene and Tetranitromethylaniline. Francisco M. Vasquez. Z. ges. Schiess-Sprengstoffw., 6, 301-3 (trans. from Memorial de Artilleria, Sept., 1910, by Hans Börner).--V. gives fully the properties of trinitrotoluene (Trilit), and the method of manuf. in Spain (sketches). The nitration of the toluene is carried out in 3 stages. For the prep. of the mononitrotoluene the waste acids from the di- and trinitrotoluenes are used. The acid is added to the toluene in the Fe nitrator and agitated, the temp. being kept below 30°. After 6 hrs. the acid is drawn off and regenerated with SO<sub>2</sub> and HNO<sub>2</sub>. The mononitrotoluene in the nitrator is then treated with the strengthened acid mixt. at a temp. of 90° for 6 hrs., the product cooled to -2° and the waste acid drawn off. A stronger acid mixt. is then added and the temp. held at 92° for 20 hrs.; then the entire contents of the nitrator is passed into a large container and allowed to stand 4-5 days, when the solid trinitrotoluene is sepd., washed, ground, and neutralized with NaOH or KOH. It is then dissolved by heating with a soln. of NaOH in acetone for 4 hrs., again solidified under water, the acetone recovered by dist., the nitro comp. washed repeatedly with hot and cold water and finally recryst. from 96% alc., the crystals being dried in drying chambers. Tetranitromethylaniline is prepared by dissolving methylaniline in 10 parts conc. H<sub>2</sub>SO<sub>4</sub>, the soln. cooled until it solidifies, and treated with 5 parts conc. HNO, at 30-40°. The product is purified in the same manner as trinitrotoluene. C. G. STORM.

Glycerol for Explosives. N. B. Stewart. Consular report for Natal. Seifensieder Ztg., 38, 987.—An attempt to use whale oil as a source of glycerol for explosives is said to be unsuccessful because of organic substances from the oil which passes into the glycerol and cause trouble during nitration.

E. SCHERUBEL.

The Moderfontein Explosion. Arms and Explosives, 19, 118.—A brief review of the report of A. B. Denne (C. A., 5, 1189) on an explosion at the Moderfontein dynamite works, Feb. 23, 1911.

C. G. STORM.

Determination of Nitric Nitrogen (PELLET). 7.

U. S., 1,000,663, Aug. 15. C. E. BICHEL, Hamburg, Ger. Assignor to E. I. du Pont de Nemours Powder Co., Wilmington, Del. Treating nitro compounds in shells by subjecting the interior of the shell containing them to air pressure sufficient to expand the shell body, and setting the explosive while under pressure. Cf. C. A., 5, 2180.

### 25. DYES AND TEXTIL CHEMISTRY.

#### L. A. OLNEY.

Process of Preparing Fuchsins and Analogous Colors. E. Suais. Sealed letter No. 1136, dated Oct. 16, 1899. Bull. soc. ind. Malhouse, 81, 180-2. Report on same. E. Noelting. Ibid., 81, 183.—In 80 g. of 66° Bé. H<sub>2</sub>SO<sub>4</sub> are dissolved 2.5 g. PhNH<sub>2</sub>; 1.8 cc. of HCHO (40%) are added with cooling and let stand overnight. Next day 7 g. of PhNHNH<sub>2</sub>HCl are added and after complete soln. the mixt. is warmed 12-15 hrs. on the steam bath. The soln. becomes brown. It is poured into H<sub>2</sub>O, pptd. with NaOH and filtered. The ppt. is redissolved in H<sub>2</sub>O and excess of HCl, salted out and filtered. The product has a dirty violet color. The author supposes first the formation of H<sub>2</sub>NC<sub>2</sub>H<sub>4</sub>CH<sub>2</sub>C<sub>4</sub>H<sub>4</sub>NHNH<sub>2</sub> which is transformed into the hydrol H<sub>4</sub>N.C<sub>4</sub>H<sub>4</sub>CH:-C<sub>6</sub>H<sub>4</sub>:NH. The latter then combines with another PhNHNH<sub>2</sub> mol. to form (H<sub>2</sub>NC<sub>4</sub>H<sub>4</sub>)2:-CHC<sub>4</sub>H<sub>4</sub>NHNH<sub>3</sub>, which by loss of NH<sub>2</sub> changes to p-fuchsine base. The possibility is indicated of preparing analogous dyes by the same reaction.

MILES R. MOFFATT.

Note on Primuline. E. Nobliting. Bull. soc. ind. Malhouse, 81, 172-3.—Hall found in 1889 that dehydrothiotoluidinesulfonic acid, which is always formed along with primuline, could be sepd. from the latter by transforming both into the NH<sub>4</sub> salts. That of primuline dissolves to the extent of forming a 20% soln. in H<sub>3</sub>O while the NH<sub>4</sub> dehydrothiotoluidinesulfonate dissolves only to the extent of a 0.7% soln. at 14.6°. The process is as follows: 100 parts of a mixt. of the sulfonic acids are suspended in H<sub>2</sub>O and satd. with NH<sub>2</sub>. After a short time the NH<sub>4</sub> dehydrothiotoluidine sulfonate crystallizes out in beautiful needles. After 2 or 3 days the crystals are filtered out. The mother liquor contains the NH<sub>4</sub> salt of primuline which is transformed into the Na salt. A sealed letter dated Nov. 2, 1889 contains a description of the process.

Formic Acid to Preserve Colors. A. SCHEURER. Sealed letter, dated Jan. 3, 1900. Bull. soc. ind. Mülhouse, 81, 153-4. Report on same. M. BATTEGAY. Ibid., 154-5. —Printing pastes which contain the mordants mixed with the color should be made so that no insol. lake is produced in the paste itself. Certain mordant colors, e. g., the nitroalizarins, have such a tendency to form lakes with metallic oxides that their use has been impracticable. When HCOOH or metallic formates are used, no preliminary pptn. in the print paste takes place. A print formula is given as follows: 250 g. nitroalizarin ( $\alpha$ - or  $\beta$ -); 65 g. Ca formate of 14½ Bé.; HCOOH 100 g.; 65 g. of Al formate 18½ Bé.; 520 g. of gum soln. A deeper shade is obtained with a mixt. of gum and starch.

Notes on the After-treatment of Dyed Cotton. M. Forr. J. Soc. Dyers Colourists, 27, 5-6.—In the case of after-treatment of sulfide dyes, these dyes contain o-quinone coloring matters, the leuco compds. of which quickly oxidize during washing off and in the air, also a varying amt. of p-quinone coloring matters, the leuco compds. of which require a long oxidizing in the air, or an oxidizing agent, to become converted into the coloring matter again. Hence the full shade is not obtained without some such oxidizing treatment. The increased fastness to light of coppered goods is due to the formation of an insol. copper lake on the fiber, by the entrance of Cu into the OH group. CH<sub>2</sub>O treatment improves only fastness to washing. Action is chem., and although not thoroughly understood, F. notes that the m-phenylenediamine group is frequently an end component of dyes recommended for CH<sub>2</sub>O treatment. Chromium fluoride is a better after-treating agent for dyed cotton than the dichromate. It seems probable that during after-chroming of cotton dyes, a dyestuff aggregate of several mols. unite with Cr to form a complex mol.

L. A. Olney.

Steam Discharge on Indigo Vat Blue by means of Chlorates, Nitrates and Bromates. Protection of the Fiber and Color. G. TAGLIANI. Sealed letter, dated April 8, 1907. Bull. soc. ind. Malhouse, 81, 156-8. Report on same. E. NOELTING. Ibid., 158-9.

—The author's proposition is to pad the cloth, before printing, with a mixt. of glycerol and gelatin, thus coating it with a hygroscopic layer which will attract moisture and thereby protect the material against the corrosive action of acid gases produced during steaming. The reviewer considers the benefit of the process small and its application limited.

MH.ES R. MOFFATT.

Tin Weighting, Fast to Light and not Weakening the Silk. O. MEISTER. Sealed letter No. 1734, dated April 22, 1907. Bull. soc. ind. Malhouse, 81, 151-2. Report on same. B. BAUER AND E. NOELTING. Ibid., 152.—HCHO and its compds., e. g., NaHSO<sub>2</sub>-CH<sub>2</sub>O, possess the same protective action as thiourea or hydroquinone and do not harm the brilliancy, feel, or shade of the silk. MILES R. MOFFATT.

The Bleaching, Thinning and Dyeing of Hair. F. J. G. BELTZER. Rev. gém. mat. color., 15, 221-5.—The steps in the process are (1) decolorizing, (2) thinning, (3) dyeing or bleaching. The decolorizing is effected by washing in a b. soln. of K<sub>2</sub>CO<sub>2</sub> (5 g. per l.), steeping 10-12 hrs. in a bath contg. per l.: 5-10 g. Na perborate, 10 g. cryst. Na silicate, 20-30 g. benzine soap. The hair is then drained, and rinsed in dist. H<sub>2</sub>O and immersed 1-2 days in a weakly alk. bath of H<sub>2</sub>O<sub>2</sub> (6 vols.). The thinning process (reducing diam.) consists in immersing the hair in Cl or Br water or in bleaching powder soln. for several days at 30°. The author divides the dyeing processes into 4 groups according to the dyes used: (1) mineral dyes, (2) vegetable dyes, (3) animal dyes, (4) coal-tar dyes. Formulas are given for the use of KMnO<sub>4</sub>, AgNO<sub>3</sub>, Pb(OAc)<sub>3</sub>, and CuSO<sub>4</sub>. If the hair is to be bleached instead of being dyed after thinning, this is accomplished using a preliminary bleach with KMnO<sub>4</sub>, SO<sub>3</sub>, and (COOH)<sub>2</sub>; and finishing with blankite.

MILES R. MOFFATT.

Use of Albumin in Dyeing and Printing. UDE. Rev. gén. mat. color., 15, 225-6.
M. R. M.

U. S., 999,785, Aug. 8. M. HESSENLAND, Höchst a/M, Ger. Assignor to Farbwerke vorm. M. L. & B., same place. Dye, 1-anthraquinonylamine-5,6-anthraquinone-thioxanthone, made by combining 1-amino-5,6-thioxanthone with  $\alpha$ -chloroanthraquinone, a red powder, difficultly sol. in the usual solvents, sol. in  $H_2SO_4$  and yielding with alk. hydrosulfite soln. a brownish violet vat which dyes cotton fast claret-red. Similar dyes are obtained by treating 1-amino-5,6-anthraquinonethioxanthone with

1,5-dichloroanthraquinone or with 1-chloro-5,6-anthraquinonethioxanthone or by treating 1-chloro-5,6-anthraquinonethioxanthone with  $\alpha$ - or  $\beta$ -aminoanthraquinone.

U. S., 999,797, Aug. 8. H. JORDAN, Leverkusen, near Cologne, Ger. Assignor to Farben. vorm. F. Bayer & Co., Elberfeld, Ger. Trisazo dye, obtainable from p-phenylenediamine, 2 mol. proportions of 1-naphthylamine-6-sulfonic acid and 1-phenyl-3-methyl-5-pyrazolone, the Na salt of the dye being a dark powder sol. in H<sub>2</sub>O with a bluish violet color and in conc. H<sub>2</sub>SO<sub>4</sub> with a green color and yielding with hydrosulfite and alkali p-phenylenediamine, 1,4-diaminonaphthalene-6-sulfonic acid and 1-phenyl-3-methyl-4-amino-5-pyrazolone and dyeing cotton reddish blue shades which when diazotized on the fiber and developed with p-naphthol change to bluer shades which are fast to light and washing and can be discharged to a pure white with hydrosulfite. p-Naphthylamine, 1-naphthylamine-7-sulfonic acid, aminonaphtholsulfonic acid, p-toluidin, cresidine, etc., may be used in making similar dyes.

U. S., 999,798, Aug. 8. G. Kalischer, Frankfort a/M, Ger. Assignor to Cassella Color Co., New York, N. Y. Dye, obtained by heating  $\alpha$ - $\alpha$ -dianthraquinonyl-1,5-diaminoanthraquinone of the formula

(obtained by condensing 1,5-diaminoanthraquinone with  $\alpha$ -chloroanthraquinone) with anhyd. AlCl<sub>2</sub>, the product being a brownish black powder, insol. in H<sub>2</sub>O and hot aniline, sol. in conc. H<sub>2</sub>SO<sub>4</sub> with a purplish blue color, pptd. from such soln. by the addition of H<sub>2</sub>O, in the form of orange flakes, and forming an orange-red vat dyeing cotton brownish orange shades fast to washing, Cl and light.

U. S., 1,000,251, Aug. 8. T. FRUSHER and T. FRUSHER, JR., Breoklyn, N. Y. Dyeing and bleaching apparatus.

U. S., 1,000,269, Aug. 8. H. JORDAN and W. NEELMEIER, Leverkusen, near Cologne, Ger. Assignors to Farben. vorm. F. Bayer & Co., Elberfeld, Ger. Blue disazo dye of the formula

a dark powder sol. in  $H_2O$  with a reddish blue color, in conc.  $H_2SO_4$  with a green color, yielding with SnCl<sub>2</sub> and HCl 2,5-diamino-4-chloro-1-anisole, 1,4-naphthalenediamine-6-sulfonic acid, and 1-naphthol-2-amino-3-sulfonic acid and dyeing cotton blue shades which when diazotized on the fiber and developed with  $\beta$ -naphthol change into a greenish blue which can be discharged to a pure white with hydrosulfite.

U. S., 1,000,313, Aug. 8. B. WUTH, Basel, Switz. Assignor to Soc. of Chem. Ind. in Basel, same place. Producing white and variegated discharge effects on colored grounds by applying to the fabric an alk. printing mixt. containing a discharging agent consisting solely of an organic ammonium compd., e. g., dimethylphenylbenzylammonium chloride, trimethylphenylammonium chloride or trimethylbenzylammonium chloride.

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- U. S., 1,000,589, Aug. 15. E. DE JOURNO, Allentown, Pa. Dyeing apparatus.
- U. S., 1,000,606, Aug. 15. M. KAHN and A. OSSENBECK, Elberfeld, Ger. Assignors to Farben. vorm. F. Bayer & Co. Trisazo dye; see C. A., 5, 3344.
- U. S., 1,000,701, Aug. 15. E. THOMANN, Zurich, Switz. Making a dense mass containing alkali peroxide (for washing, bleaching, etc.) by m. 12 parts KNO<sub>2</sub>, mixing therewith 10 parts of the peroxide, and breaking up the mass thus formed when cold. Where a neutral reaction of the soln. is required, the compd. may be plunged into molten nitrate to which KHSO<sub>4</sub>, Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> or H<sub>2</sub>BO<sub>3</sub> has been previously added to form a coating on the pieces.
- U. S., 1,000,899, Aug. 15. C. DE LA HARPE and R. BURCKHARDT, Basel, Switz. Assignors to Dye-Works formerly L. Durand, Huguenin & Co., same place. Making leuco dyes of the gallocyanin series by causing an aralkylamine, e. g., benzylamine, to react with a gallic acid gallocyanin, decarboxylating the resulting condensation product and finally converting the product into a leuco deriv. by treating it with the usual reducing agents.

## 26. PIGMENTS, RESINS, VARNISHES AND INDIA RUBBER.

A. H. SABIN, THEODORE WHITTELSEY.

Advances in the Manufacture of Ultramarine. M. VON REIBOLDT. Chem. Ztg., 35, 1023-4.

Manufacture of White Lead by the Mild Process. FABIO VILLANI. Ind. chim., 11, 153-4.—Brief description of the process. Chas. A. ROUILLER.

Amber Substitutes and their Recognition. J. MARCUSSON AND G. WINTERFELD. Kunststoffe, I, No. 15; through Chem. Rev. Fett-Harz-Ind., 18, 224.—Method to distinguish copal from amber: 2 g. of the finely powdered sample are heated under the reflux condenser with 25 cc. cajuput oil for 10 min. After cooling, filter through a folded filter and add to the filtrate 2-3 vols. of heavy benzine. If copal is present a distinct ppt. is observed, if amber only a light cloudiness is shown. Hardened copal is used as a substitute for "pressed amber," made by pressing small bits of amber into larger masses. This hardened copal is recognized by its low ester no. 2.5-8.5; ester no. of amber is 71-112. Hardened copal is also nearly free from S; amber shows 0.34-0.42% S. All of the above tests refer to unmelted amber.

Industrial Applications of Naphthenic Acid. N. Chercheffsky. Caoutchouc and gutta-percha, 7, 4574-9.

J. J. M.

Machines for the Manufacture of Caoutchouc. P. BREUIL. Caoutchouc and gutta-percha, 8, 4742-5, 4845-6, 4917-22, 5128-36, 5331-5.

J. J. M.

Application of Artificial Cooling in the Caoutchouc Industry. J. BOUTARIC. Caoutchouc and gutta-percha, 8, 496-7.

J. J. M.

Dynamometry of Plastic Materials. L. CLEMENT AND C. RIVIÈRE. Caoutchouc and gutta-percha, 8, 5156-7, 5316-26. J. J. M.

The Preparation and Industrial Value of the Different Kinds of Raw Caoutchoucs.

G. Vernet. Caoutchouc and gutta-percha, 8, 5281-6, 5500-7.

J. J. M.

Cultivation of Rubber Plants. E. DE WILDEMAN. Caoutchouc and gutta-percha, 8, 5028-32; Rev. gén. sci., 22, 32-7.

J. J. M.

Propositions for a Uniform Execution of Tests in the Evaluation of Caoutchouc.

Anon. Caoutchouc and gutta-percha, 8, 5011-23.

J. J. M.

Viscosimeter (STORMER). 1.

- U. S., 999,755, Aug. 8. X. Debedat, Bordeaux, France. Obtaining coloring matter from plants, e. g., heather, pine bark, oak bark or chestnut, containing tannin, by b. in H<sub>2</sub>O to ext. the tannin, then b. in an alk. soln., e. g., Na<sub>2</sub>CO<sub>2</sub>, adding an acid to the soln. to obtain a ppt. and washing and drying the latter. "Heather carmine" thus obtained is insol. in cold H<sub>2</sub>O, alc., ether, CHCl<sub>2</sub>, benzine, etc., and but slightly hygroscopic in either acid or alk. state.
- U. S., 999,819, Aug. 8. J. MARKUS, Manchester, Eng. Forming articles (e. g., boot heels) from waste rubber, by forming a blank by cutting a flat strip from the waste, shaping and drilling the blank and subjecting it to pressure and a heat below the original vulcanization temp. to give a superficial finish.
- U. S., 1,000,162, Aug. 8. C. ELLIS, Montclair, N. J. Assignor to Chadeloid Chem. Co., New York, N. Y. Paint or varnish remover, comprizing about 40 parts of a 50% aq. soln. of K cresylate, 20 parts solvent naphtha in which 2 parts of ceresin have been dissolved, pine oil 10, benzyl alc. 20 and Turkey red oil 5 parts. Other formulas are given including paraffin, Na carbolate, benzene, pine oil, rosin, cresylic acid, KOH, K oleate and chlorocresol.
- U. S., 1,000,388, Aug. 15. C. Ellis, Larchmont, N. Y. Finish remover containing C<sub>4</sub>H<sub>6</sub> 12 gals., hexahydrobenzene 27 gals., com. adipin ketone 43 gals., wood flour 50 lbs. and paraffin 30 lbs.
  - .U. S., 1,000,405, Aug. 15. C. J. HEALY, Brooklyn, N. Y. Varnish filter.
- U. S., 1,000,634, Aug. 15. GRAY STAUNTON, Evanston, Ill. Printer's roll formed of a vulcanized base comp. of pontianak 9, vegetable oil, e. g., cottonseed oil, 4, and Ca(OH)<sub>2</sub> 2 parts, mixed with a filler of pre-vulcanized rubber, wool grease, S and lime and then vulcanized.
- U. S., 1,000,781, Aug. 15. A. T. COLLIER, St. Albans, Eng. Flexible composition consisting of an intimate mixt. of vulcanized India rubber and individual short lengths of thread composed of fibers tightly twisted together. For heavy pneumatic tires 20-30% of cotton threads are combined with the rubber.
- U. S., 1,000,943, Aug. 15. H. C. PRITCHARD, Colfax Twp., Mecosta Co., Mich. Paint (for wood, metal, stone or cement) formed from white lead or ZnO 400 lbs., slag from Fe smelting 267 lbs., 30 gals. of aq. liquid from b. rice 25 lbs., shellac 15 lbs., potash 2 lbs., H<sub>2</sub>O 10 gals., rosin 25 lbs., gasoline 10 gals. and boiled linseed oil 50 gals.
- U. S., 1,000,966, Aug. 15. CARLETON ELLIS, Larchmont, N. Y. Composition for cleaning paint, varnish, etc., comprizing soap, H<sub>2</sub>O, linseed oil and sulfonated oil, the amt. of linseed oil being sufficient to more than satisfy the alkali set free by hydrolysis on dilution.

## 27. FATS, FATTY OILS AND SOAPS.

#### E. SCHERUBEL.

The Use of Poisonous Cardamom (Maratti) Fat in the Preparation of Margarine. H. THOMS AND FRANZ MÜLLER. Berlin. Z. Nahr. Genussm., 22, 226-36.—The chem. consts. of chaulmugra, and crude and refined "maratti" fat, are so similar that they can hardly be distinguished by such means. As the first is only a slight purgative

and the latter a poison, it is evident that physiological, as well as chem. tests should be made with new fats, before they are used for human food. H. S. BALLEY.

Chemical Examination of the Oil from Seeds of Bursaria spinosa. (Blackthorn) E. GRIFFITHS. J. Proc. Roy. Soc. N. S. W., 43, 399-405.—The seeds yield 17% of oil to petroleum ether. The constants are: d<sub>12</sub> 0.8867, w at 21° 1.4681, free acid 1.26, I no. 86.4, sapon. no. 169.5. The chief constituent of the oil is oleic acid and small amts. of solid acids (among which is stearic) are also present. Phytosterol was detected in the unsapon. portion and also a wax-like substance m. at 45°.

E. SCHERUBEL.

Siver, a New Oil Seed from Sumatra. H. Gorter. Seifens. Ztg., 38, 987.—This oil comes from Xanthophyllum lanceolatum, Boerl. The seeds contain 8.9% H<sub>2</sub>O, 39.17% fat, 2.42% ash, 6.32% crude fiber, 5.4% protein, 37.75% N free extractives. The recovered oil is colored green by chlorophyll. It solidifies partly at 30° and completely at 15°; m. p. of fat 48°, of fatty acids 54°, free acid 12.2%, sapon. no. 198.5, R.-M. no. 0.0, I no. 36.6, mean mol. wt. of fatty acids 268. Siver fat is similar to tangkawang fat and can be used in the candle and soap industry. It contains a poison, a saponin, which remains in the press cake. The oil is not poisonous and is used as an edible fat by natives.

Soy-bean Oil. H. MATTHES AND A. DAHLE. Arch. Pharm., 249, 224-35.—The oil contains 94% fatty acids in the form of glycerides, 15% as palmitic, 80% oleic, linoleic and linolenic. Free fatty acids are present in but small amt. The pure oil has d. 0.9260 at 15°, solidification point —11.5°, \*\* 1.468, (α) 0, acid no. 5.711, ester no. 186.589, sapon. no. 192.3, Hehner no. 94.07, I no. 131.3, R-Meissl no. 0.7549, Polenske no. 0.7843, Elaidin test positive.

The Phytosterol of Soy-bean Oil. H. MATTHES AND A. DAHLE. Arch. Pharm., 249, 436-44.—The unsaponifiable portion of the oil amts. to 0.7%, consisting of almost equal parts of solid and liquid substances. The solid contains 2.4% of a *l*-phytosterol, m. 169°, identical with the stigmasterol isolated from the oil of calabar bean, and 97% of a *l*-phytosterol, m. 139°, with one double bond. The liquid portion consists of unsatd., O-containing compds. giving phytosterol reactions, the elementary analysis being the same as for the second phytosterol above. Expts. with digitonin to det. phytosterol addition products contained in the liquid portion were without results.

H. C. Fuller.

The Soy Bean. H. VAN DER WARRDEN. Royal Museum. Pharm. Weekblad, 48, 889-96.—Review of the literature in regard to the bean, as food for man and animals and technical value of the fat, with some new analysis of the bean showing high protein (35.4%) and fat (17%) content; physical constants of the oil and of the fatty acids; analysis of the meal left after oil extraction; protein (40.5%), fat (5.8-6.25%). Its ash has a high  $P_2O_4$  value (29-36%).

Rapidity of Solution of Soaps in Water. A. A. Zhukov and P. I. Skestakov. Seifens. Zig., 38, 982-3.—The solubility depends upon size and shape of soap particles, temp. and conc. of the soln. and mechanical condition of soln. formed. The app. used consisted of a cylinder, such as is used for drying gases, with an inlet at the bottom and a rubber stopper at the top with an outlet and a thermometer. The cylinder is half filled with glass beads which support a wire gauze upon which 1 g. of the soap is placed. Water at 45° is allowed to flow through at the rate of 200 cc. per min. and the time noted for the soap to dissolve. The results show that rapidity of soln. depends mainly upon the character of the fats and that the H<sub>2</sub>O content is of secondary importance. The results for various curd soaps showing time in min., % of pure soap

and nature of fats resp., are as follows: (1) tallow soap, over 480, 64.8, tallow; (2) tallow and rosin, 147, 67.0, tallow and 20% rosin; (3) tallow and coconut oil, 127, 66.5, tallow and 10% coconut oil; (4) millet toilet soap, 108, 85.3, tallow, cottonseed and coconut oil; (5) tallow olein rosin soap, 80, 86.9, tallow, olein, rosin; (6) tallow and rosin, 60, 65.0, tallow and 30% rosin; (7) tallow and coconut oil, 53, 65.2, tallow and 25% coconut oil; (8) curd soap, 52, 62.8, tallow, cottonseed oil and rosin; (9) curd soap, 42, 78.1, tallow, cottonseed oil and rosin; (10) curd soap, 42, 72.0, tallow, cottonseed oil and rosin; (11) curd soap, 41, 64.2, 65% palm kernel, 20% C. S. oil and 15% rosin; (12) curd soap, 35, 70.4, 62.5 palm kernel, 22.5% peanut and 15% rosin; (13) Marseilles soap, 34, 68.5, coconut and cottonseed oil; (14) curd soap, 30, 69.0, 75% palm kernel and 25% rosin. For gelatinous soaps: (15) Russian coconut oil soap, 70, 50.0, tallow and 25% coconut; (16) same, 43, 55.1, tallow, coconut and C. S. oil; (17) same, 40, 50.5, same; (18) German coconut oil soap, 20.5, 56.7, palm kernel oil; (19) mottled coconut, 10.0, 35.4, coconut oil; (20) cold made coconut soap, 9.0, 27.3, coconut oil. E. SCHERUBEL.

Constitution of Soap Solutions. J. McBain and M. Taylor. Seifens. Zig., 38, 986; see C. A., 5, 2360. E. S.

Valuation of Cresol Soap Solutions. H. Seiger. Apoth. Ztg., No. 37; Seifens. Ztg., 38, 986.—Detn. of jatty acids: 20 g. of soln. are evapd. to dryness twice with 500 cc. H<sub>2</sub>O to drive off cresols. The residue is transferred with 40 cc. H<sub>2</sub>O to a 160 cc. grad. cylinder and shaken with 5 g. NaCl and 10 cc. HCl (sp. gr. 1.19) and then 20 cc. petroleum ether added. The ether soln. is read off and 20 cc. subtracted, the diff.—the fatty acids which × 0.92 — wt. in g. Detn. of cresols: 20 g. of soln. are dild. with 20 cc. H<sub>2</sub>O and treated exactly as above without previous evapn. The upper layer consists of cresols, fatty acids and ether, from which the vol. of petroleum ether and fatty acids detd. above is subtracted and the remainder × 1.04 — wt. of cresols in g.

Measurement of Density of Soap Solutions. E. CORNISH. Seijens. Ztg., 38, 986; see C. A., 5, 2360. E. S.

The Stearin Content of Paraffin Candles. J. MARCUSSON. Chem. Rev. Fett.-Harz-Ind., 18, 210-1.—The current statement in text-books that 5-15% stearin is added to paraffin candles to harden them is contradicted by saying that only 1% is added during the winter and not more than 2% during the summer, and only for the purpose of preventing their adherence to the molds. To distinguish paraffin from petroleum and from brown coal, the I no. is used, the former showing an I no. under 12 and the coal-paraffin above 18.

P. ESCHER.

Peanut Oil in Olive Oil (FACHINI, DORTA). 12.



U. S., 1,000,487, Aug. 15. J. W. BLAGDEN, Mannheim, and R. MÜLLER, Eilenburg, Ger. Assignors to C. F. Boehringer & Soehne, Mannheim-Waldorf, Ger. Preparing fatty acid compounds, e. g., for making candles, by dissolving a fatty acid in a hydrocarbon, e. g., oleic acid in petroleum, cooling the soln., treating it with NH<sub>3</sub> and subjecting to pressure and drying in vacuo at a temp. below 100° to remove the excess of solvent and NH<sub>3</sub>.

U. S., 1,000,675, Aug. 15. A. W. FRENCH, Piqua, Ohio. Apparatus for cooking oil-bearing material, e. g., cotton seed.

### 28. SUGAR, STARCH AND GUMS.

#### A. HUGH BRYAN.

The Presence and the Determination of Nitrates in Cane and Beet Molasses. H. PELLET AND CH. MÜLLER. Intern. Sugar J., 13, 493.—Expts. demonstrated that cane molasses contains nitric N. It was found impossible to use Schlösing's method and the following modification of that of Boussingault was finally adopted: Take 20 g. of molasses and 40 g. of pulverized sieved pumice stone. Mix well in a Ni capsule, add a little b. H<sub>2</sub>O, and dry in oven. Weigh, remove the residue and weigh the capsule. Exactly half this residue, corresponding to 10 g. molasses, is weighed out, ground and placed in a 300 cc. flask with 200 cc. 80% alc. Let stand 12 hrs., with frequent shaking, and filter. From 100 cc. of filtrate expel the alc. Connect to the Schlösing app. and det. N in the ordinary way. Pozzi-Escot's method (C. A., 4, 561) may also be used. Connect flask from which the alc. has been expelled with the ordinary app. for detn. of NH<sub>2</sub>, first adding (1) 5-6 g. fine Al clippings, not Al powder; (2) 2 cc. of a conc. soln. of HgCl<sub>2</sub>; (3) 100 cc. dist. water; (4) 2-3 pieces Zn to aid boiling; (5) a little vaselin. A brisk reaction soon begins, especially if warmed. The outlet of the condenser dips into 20 cc. of 35% H<sub>2</sub>SO<sub>4</sub>. When the reaction has ceased, add NaOH (35° Bé.) through a funnel in the stopper of the flask, heating meanwhile to avoid absorption and to expel all the NH<sub>a</sub>. After 2/3 of the liquid are distilled, add a few cc.of Na<sub>2</sub>S<sub>2</sub>O<sub>2</sub> to break up NH<sub>4</sub> mercuric compds. Titrate the distillate with NaOH of the same strength as the acid. These 2 methods agree very well, and both show about 0.5% N calc. as KNO.

The Deposit which Forms on Diluting Molasses. H. Peller. Interm. Sugar J., x3, 500.—Exam. of the deposit which forms and settles out on diluting molasses shows that it is substantially of the same nature as that which forms in the evapg. app. A study of the conditions under which molasses is produced show that this deposit is due almost entirely to imperfect clarification, since juices completely clarified by an excess of CaO under the proper conditions give a molasses which does not produce this deposit. Molasses may be tested for clarification as follows: 100 g. are diluted with 1000 cc. dist. water and a few cc. of a soln. of Ca sucrate added; after heating to b. a more or less abundant ppt. is produced. This may be collected, washed, dried, and weighed, and the mineral matter in it detd.

A. Given.

Results of Clarifying Tests with "Eponit." H. C. PRINSEN GERRLIOS. Arch. Swikerind., 19, 1085-6.—The author quotes Spakler to the effect that if the cost of clarifying with bone-black be taken as 1, clarifying with "eponit" will cost 1.7, and with "blankit," 2. "Eponit" is not suitable for white sugar manuf. as it is so fine that it passes through all types of filters and gives a turbid thick juice with correspondingly poor-colored sugars. The same results were obtained with a very fine C produced in the manuf. of KCN from K<sub>4</sub>Fe(CN)<sub>8</sub>, which had a very high decolorizing power but could not be filtered out.

W. L. Badder.

The Capability of Sugar Beets to Take up Arsenic. H. REMMLER. Chem. Zig., 35, 977-9.—Expts. show that the sugar beet possesses the power to take up As. The quantity absorbed increases with the quantity of Schweinfurter green applied to the beets. Whether As is absorbed through the leaves or through the beets or both, remains to be worked out, as well as whether it is combined with the CaO and Fe or some organic As compd. is formed. Even in the case that As is stored in the beet in its most poisonous form, the fears of critical experts are discredited. It was not possible to find As in appreciable quantities in beet leaves which have been sprinkled with Schweinfurter green. Considering the mfg. processes of sugar and the great tenacity

A., 5, 2442.

of As for lime it would seem that any As that might get into the sugar would be removed in the defecation and there would be more danger of introducing As through the burned lime and milk of lime than through the beets.

A. H. BRYAN.

The Deterioration of Raw Sugars in Storage. F. E. Combs. Louisiana Planter, 47, 201-2.—C. refers to the work that has been accomplished along this line in way of bacterial decomp. but believes an important consideration has been left out, vis., the temp. of the sugar on bagging. "If the sugar is dried hot, as much of it is, and tied into the package at a temp. much above that of the air, then the exhaling moisture will condense upon the cool surface layer under the sacking until there is a skin of wet sugar just under the jute freely exposed to the spores of all manner of ferments and damp enough to start a quick growth of any of them." If the outer layer of sugar is no cooler than the rest, no condensation takes place and, hence, less chance for decomp. A. H. B.

Calculation of the Values for Rating the Performance of Cane Mills. F. W. Bol. K. Arch. Suikerind., 19, 842-59, 871-94, 903-31.—Mathematical and very voluminous.

W. L. BADGER.

Cleaning the Steam Pipes of an Evaporator with Benzine. A. FRANKEN. Arch. Swikerind., 19, 1224-5.—The steam pipes of a vertical evaporator were filled with water, a layer of benzine poured on this, and a cock in the bottom opened so that the liquids were 2 months in running out. Very good results were obtained. W. L. BADGER.

Wet and Dry Bagasse as Fuel. Erkelens. Arch. Suiterind., 19, 949-57.—The theoretical amt. of steam produced is increased 22% by drying bagasse from 48% to 12% H<sub>2</sub>O. The heat of the waste gases is nearly sufficient for this.

Wet and Dry Bagasse as Fuel. A. FRANKEN. Arch. Suikerind., 19, 1215-20.

W. L. B.

A. H. BRYAN.

W. L. B.

Dried Bagasse as Fuel. KERR. Eng. News, 66, 305; see C. A., 5, 2198; cf. C.

B. GRIVEL, Arch. Suikerind., 19, 1221-3.—Discussions (preceding abstr.).

The Projects of the Experiment Station. R. E. BLOUIN. Rev. ind. agr. Tucumon, 2, No. 1, 5-16.—A brief review of the important results relating to the sugar industry covering expts. on: Fertilizers, cultivation, in Hawaii; irrigation; loading machines; and varieties of cane. The review also covers entomological, pathological, bacteriological and chem. work.

G. A. ROUSH.

Sugar Products and Confections in the Austrian Alimentary Codex. Anon. Oesterr.-Ung. Z. Zuckerind., 40, 631-67.—Methods for the analysis of raw sugar, molasses, com. grades of sugar, sirups, starch sugar, maltose sirup, levulose, sugar colors (caromel), milk sugar and of confections are given.

A. H. BRYAN.

Analytical Work of the Austrian Sugar Experiment Station. Anon. Oesterr.-Ung. Z. Zuckerind., 40, 604.—Results of the analysis of 26 samples of limestone, 3 of lime, 16 of coal and of the chem. exam. of some 50 samples of cattle foods are given.

The Story of the Russian Sugar Industry. Indische Mercuur; Intern. Sugar J., 13, 479.

A. GIVEN.

The Air-nitrogen Fertilizers, their Preparation and Importance for the Java Sugar Industry. C. J. Millo. Arch. Suikerind., 19, 1053-75, 1093-1134.—M. describes the processes for the manuf. of nitrogenous fertilizers from atmospheric N, as nitrates, NH<sub>4</sub> salts, Ca cyanamide, etc., and compares the cost and availability of the N in the different products.

W. L. Badge.

What Procedure Must be Followed in Beet Culture to Secure Sound Beets and Certain Yields? K. STÖRMER. Deut. Zuckerind., 36, 403-8.—Expts. are cited to show that in dealing with the root-blight, proper fertilizing, favorable weather, and rapid growth, are more important than sterilizing the seed. Seed soaked in 1/2% phenol gave the same yield and little less root-blight than untreated seed. Complete fertilizer, (N, P, K, Ca) gave over twice the yield and only half the no. of blighted plants. Both K and Ca are necessary to prevent the root-blight. Additions of NaCl or CaSO<sub>4</sub> to the complete fertilizer gave a yield over 3 times that with N alone. The omission of P has little effect. As the beet grows best in its wild state, i. e., in salt marshes it is necessary to fertilize heavily with NaCl, KCl and CaSO<sub>4</sub>. With reference to "beet-tired" soils, the parasite Heterodera schactus is commonly held responsible. Attempts to combat this disease by the above principles were unsuccessful; and as the infection is from the soil and not from the seed, sterilizing the seed is useless.

W. L. B.

Influence of the Soil, Culture and Fertilizers, on the Appearance of Root-blight, Heart and Dry Rot. Schander. Deut. Zuckerind., 36, 446-7.—Root-blight is held to be due rather to mechanical soil conditions than to infected or impoverished soils. It tends to appear least in loose soils, and oftenest in heavy soils, especially in such as tend to cake. The cause of heart and dry rot is as yet unknown, but it is connected in some way with weather and soil conditions. Deep working of the soil, is recommended for both diseases.

W. L. B.

Cultivation and Manufacture of Cane Sugar in the Hawaiian Islands. Alfredo Ferreyos. Rev. ind. agr. Tucuman, 2, No. 1, 31-46.—Describes climatic conditions, soils, labor, administration, cultivation, varieties of cane, irrigation, cutting, fertilization, experimental fields, insect pests.

G. A. ROUSH.

Asexual Selection of Sugar Cane in Java. J. SIBINGA MULDER. Intern. Sugar J., 13, 473.—After years of expt. it has been decided that selection of sugar cane for planting along the lines of greatest sugar content or greatest wt. of stalks or tops fails to consistently improve the yield and sugar content of cane, and that the only practical method is that of natural selection.

A. GIVEN.

The Effect of the Treatment of the Seed on the Development of the Sugar Beet. SCHANDER. Deut. Zuckerind., 36, 443-4.—The only practical method of treating beet seed is hulling. Soaking, disinfecting, etc., are either of no use or too expensive.

W. I. RADGER.

Gum and Gelatin. F. W. TIEBACEX. Pharm. Weekblad, 48, 691-3.—The coagulum (T. admits this is not an accurate use of the word) obtained by the pptn. of a gum-gelatin soln. is opaque, jelly-like and white, differing thus from a jelly due to soln. of equal quantities of the 2 substances. They also react differently to various chemicals, e. g., with basic Pb(OAc)<sub>2</sub>, the coagulum becomes at once milk-white and in 1 day is hard; the jelly after 1 hr. is cloudy, after 24 hrs. still soft. With Na<sub>2</sub>SO<sub>4</sub> the coagulum swells. The coagulum seems to consist of drops of gelatin coated with gum; the jelly is a soln. of gum in gelatin. The coagulum does not adhere to filter paper until it becomes dried, when, due to loss of acid, the adsorption combination, acid + gum-gelatin, breaks down and the filter absorbs the gum. This combination may be broken down by neutralization, i. e., addition of salts or of more acid. In salts, KOH or weak acids the coagulum swells; the swelling is reversible. The acid adsorption varies with the quantity of gum and inversely with the temp. V. E. HENDERSON.

Determination of Sugar in the Blood (FORSCHBACH, SEVERIN). 11. Sugar in Urin (DE JAGER). 11.



How does Cane Make Sugar? (CHAVANNE) 11. Maize Studies (HANAUSEK). 12.

U. S., 1,000,726, Aug. 15. E. O. ECKLAND, Keokuk, Iowa. Making starch by soaking grain in a satd. soln. of O<sub>20</sub> grinding the grain and removing the germ from it, reducing the material to a pulp and sepg. the starch milk from it.

### 30. PATENTS.

Abstracts of patents are included under the foregoing divisions of the Journal. The abstracts of U. S. Patents are prepared by Earl T. Ragan, those of the British, French and German Patents by O. D. Swett and those of Canadian Patents by Russell S. Smart, of Fetherstonhaugh & Co., Ottawa (5 Elgin St.). The abstracts of French Patents are prepared, with permission, on the basis of the abstracts in Moniteur scientifique, those of German Patents, with permission, on the basis of the abstracts in Chemisches Zentralblatt.

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# CHEMICAL ABSTRACTS

Vol. 5. DECEMBER 10, 1911.

No. 23

#### I. APPARATUS.

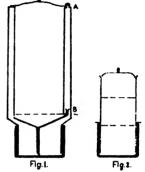
L C. IONES.

A Simple Steam Sterilizer with Attachment for Converting to a Drying Oven. B., RUPP. A poth. -Ztg., 26, 75.—A double-walled cylindrical vessel forming a steam

jacketed chamber closed by a cover and fitting into an iron vessel in which the steam is generated. By closing A the app. may be used for sterilizing in streaming steam, by closing B with a stopper inserted on a rod through A, it becomes a steam jacketed drying oven. Fig. 2 shows a single walled vessel designed for steam sterilization.

A. SEIDELL.

Apparatus for Sulfur Determination in Iron and Steel. D. A. Wennmann. Chem. Ztg., 35, 863.—Two pieces of app. without rubber connections are described. The first has a single ground joint in the neck of the flask for attaching the cooler which consists of a cylindrical vessel, open at the top, through which the cooling tube passes to the absorber. The HCl is poured in the cooler and by



slightly turning the ground joint runs into the flask through a channel in the neck, after which the joint is turned back and the cooler filled with H<sub>2</sub>O. There is a side tube for admitting CO<sub>2</sub> when necessary. In the second app. a funnel tube is passed down through a Liebig cooler. A 2-way cock and side-tube below the funnel admit HCl or CO<sub>2</sub> as desired.

J. H. Moorr.

A New Apparatus for Generating Gases. LÉGER. Chem. Ztg., 34, 1061.—An app. made up of a glass cylinder, containing a glass tripod on which the glass receiver for the Zn, etc., is set.

E. J. WITZEMANN.

Precision Fermentation Saccharometer. Anon. Chem. Ztg., 35, 871.—Söhle's saccharometer, consisting of a Hg reservoir connected at the bottom with a vertical graduated tube. A neck on top of the reservoir carries a hollow glass stopper inclined at an angle of about 45°, small end up, a hole in one side of the stopper communicating with the reservoir through the neck. The sugar soln. and yeast are placed in the stopper, the evolved CO<sub>2</sub> forcing the Hg up the graduated tube, the readings of which give the sugar content direct.

J. H. Moore.

An Apparatus for the Determination of the Melting Points of Fats. L. V. LIEBE-MANN. Hyg. Inst., Budapest. Z. Nahr. Genussm., 22, 294-5.—The bend of a "U" tube is filled with Hg and into 1 arm the melted fat is poured. The tube is then closed with a stopper carrying the thermometer, a Pt contact wire and a small funnel, the thermometer and wire ending about the middle of the fat layer. After the fat has properly set, a layer of Hg is run in through the funnel, and the tube clamped in place on a bath and heated. A 2nd wire inserted in the Hg in the open end of the "U" tube is attached through a battery and bell to the first, and when the fat m., the upper layer of Hg runs through it, completing the circuit and ringing the bell.

H. S. BAILEY.

Apparatus for Liquefying Hydrogen. NERNST AND POLLITZER. Univ. Berlin. Z. Elektrochem., 17, 735.—The app. is constructed on the Linde principle. It is so designed that reactions can be carried out in 1 of the compartments. A small Dewar flask fits inside of a thin walled brass "test-tube" closed at the top with a cork. The brass tube is set into a larger Dewar into which liquid air is introduced until the cork of the brass tube is well covered. The H gas under a pressure of 150 atm. is passed through a long double walled Cu spiral, partly immersed in the liquid air, the Linde principle of precooling being used. With a H flow of 2 to 3 cm. per hr. 10% is liquefied and at the same time only 0.331 of liquid air is evapd. There is no danger of explosion providing the temp. and pressure of the liquid H is regulated. Only pure H must be used. The temp. of the liquid H measured with a Cu-constantan couple corresponds to 5.3 volts compared to 6.0 volts for liquid air. It is manufd. by Hoenow Bros., Berlin, 0.112. Price, \$60.00.

C. G. Fink.

Smoke	Abatement	(Owens).	21.	
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- U. S., 1,001,924, Aug. 29. J. BOLAND, Decatur, Ill. Acetylene generator.
- U. S., 1,003,047, Sept. 12. L. R. IRUZ, Mexico, Mexico. Carbide holder for acetylene generators.
- U. S., 1,003,594, Sept. 19. J. Geistdörfer, Paris. France. Apparatus for extracting albumins from plants, comprizing a series of closed extraction receptacles, connections for circulating solvent therethrough under partial vacuum and condensers for recovering vaporized solvent.
- Brit., 8,582, Apr. 9, 1910. F. J. TURQUAND, 4 and 5 Albany Court Yard, Piccadilly, London, W. In an app. for the detection of dangerous gases, a device sensitive to the diffusion action of gases, said device comprizing 2 chambers that only communicate with the exterior by porous septa, a pressure indicator communicating with the chambers and means for closing or unclosing the porous septa and preventing or permitting any diffusion therethrough.

Brit., 16,194, July 6, 1910. T. C. PALMER, 98 Commercial Road, East London, E. In app for collecting carbon dioxide from fermentation backs and compressing same, the use of a chamber located intermediate of the back or backs and the compressor and having a flexible wall or part to be displaced or distended by, and to an extent more or less according to the pressure of the gas within the chamber, the movement of the flexible wall or part being utilized to regulate the area of the outlet from the chamber to the compressor.

Brit., 18,199, July 30, 1910. H. O. CARR, 26 West Side, Wandsworth Common, London, S. W. App. for testing the illuminating power of gas as it comes direct from the exhausters, comprizing a condenser and a purifier combined with a photometer.

### 2. GENERAL AND PHYSICAL CHEMISTRY.

JOHN JOHNSTON.

Fundamental Properties of the Elements. T. W. RICHARDS. J Chem. Soc, 99, 1201-18. (Faraday Lecture for 1911.)—The importance of accurately detg. the values of the at. wts., compressibilities, and heats of reaction is emphasized and the methods employed in precise detns. are described briefly. The hypothesis of compressible atoms and its ramifications are discussed and shown to be in harmony with the experimental results. All of these properties are shown to be periodic, but with periods varying in length.

J. Hunt Wilson.

The Connection between the Volatility, Fusibility and Density of Compounds, and the Chemical Forces at Play within their Molecules. Geoffrey Martin. Chem. News, 104, 29. Comments on Richards' Faraday Lecture (preceding abstr.).—M. in 1905 published the generalization that it is "the internal chem. forces which the ats. exert on each other in the mol. which decide the internal attractions with which the mols. themselves are attracted together and consequently properties arising out of this mol. attraction such as volatility, fusibility, hardness, and d. of the compd."

George W. Morey.

The System of the Elements on the Basis of the Periodic Dependence of the Vibration Number and the Atomic Weight. W. BILTZ, Clausthal, Z. Elektrochem., 17, 670-4.—B. has calc. the vibration const. v, for all the elements from the equation of Lindemann,  $\nu = 2.12 \times 10^{12} \sqrt{T/mv^2/s}$  where T is the abs. m. p., m the at. wt., and v the at. vol. (C. A., 4, 2593). In the case of allotropic elements,  $\nu$  was calc. for the form stable at the m. p. The results are tabulated and also plotted, using  $\nu$  as ordinates and the at. wt. as abscissas. "The resulting curve allows a deeper and more comprehensive insight into the relationship of the elements than one has hitherto known." Homologous elements are found in corresponding positions on the curve. Dissimilar elements which are grouped together in the Mendelejeff table are here sharply differentiated, e. g., Cs and Au. H falls in the halogen group. Hg is grouped with Ga and In. C occupies a unique position with the highest vibration no. 21,10<sup>12</sup> for graphite to 24.10<sup>12</sup> for diamond. The elements with high valence form the maxima and the argon gases the minima, which was to be expected in the light of J. J. Thomson's at. model, since an element with a relatively rapidly rotating electron ring means one with a relatively high value of v. J. L. CRENSHAW.

Constitution of the Atom. HAROLD A. WILSON. Proc. Am. Phil. Soc., 50, 366-73.—The object is to develop a theory of the constitution of the atom, susceptible of numerical verification. As a foundation for further calculation, W. adopts the view of J. J. Thomson that the atoms are rigid spheres of + electricity containing - electrons which can move about freely through the + charge. The question as to the number of electrons contained in an atom has already been answered approx. by examg. the effects of matter on light and Röntgen rays. From such considerations as these, J. J. Thomson reached the conclusion that the no. of electrons contained in an atom is of the order of the at. wt. of the atom in question. For example, Crowther finds as a result of experiments on the energy scattered when Röntgen rays pass through a plate of Al, that the no. of electrons per atom of this element is 85, which is about 3 times the at. wt. Since the mass of the electron is but 1/1700 of an H atom and since the no. of electrons per atom is comparatively small, it is evident that the electrons contribute but a negligible part to the total mass of the atom. In his original theory, J. J. Thomson assumes that the electrons are arranged in concentric spherical shells within the positive sphere. Upon making the attempt to show that the chem. behavior of the elements could be accounted for by considering the no. of electrons available and their distribution it was found that the solution was attended by insuperable difficulties. W. shows that in order to det. approx. the distribution of n electrons in the + sphere it is sufficient to find out how the sphere may be divided up into \* equal vols., as nearly spherical as possible, and to put an electron at the center of each of these. If n is large, the electrons are arranged like the centers of the shot in a pile of shot. It is assumed that these elementary vols. are arranged in spherical layers and that, in the case of the atom in the same group of the periodic system, one atom is formed from another by the addition of another spherical shell of elementary spherical vols. From simple considerations the relation is deduced that B = 4n/3c where B is the proportionality factor between the at. wt. of an atom and the no. of electrons

it contains and c is the difference of the cube roots of the at. wts. of 2 successive elements in the same group. The mean value is c is 0.81, hence B=8. The final relation to be tested takes the form:  $A_m=(A_1^{1/6}+(m-1)(4\pi/3B)^{1/6})^8$ .  $A_m$  is the at. wt. of the mth element in a given group,  $A_1$  being that of the 1st element of the group. From this it is evident that if the at. wt. of the 1st of a series of similar elements be known, the others may be calc. by substituting for m the integers 1, 2, 3, etc. A typical instance is cited: Values of m from 1-6 correspond to values of  $A_m$  as follows: 1, (H=1); 6, (Li=7); 18, (Na=23); 40, (K=39); 77, (Rb=85); 129, (Cs=133). Since the calculations lead to results which are so well in accord with observations, the theory must be looked upon as containing a considerable element of truth.

A. H. PFUND.

Investigation of the Determinations of the Law of Chemical Attraction between Atoms from Physical Data. R. D. KLEEMAN. Phil. Mag., 21, 83-102.—K. investigates to what extent the law of chem. attraction can be derived from latent heat, surface tension and other physical properties of substances; and, on the basis of the assumption that the attractions between mols. varies inversely with the fifth power of their distance apart, gives an empirical formula which correlates the above quantities.

ROBT. F. EARHART.

Heat of Mixtures of Substances and the Relative Distribution of the Molecules in the Mixture. R. D. KLEEMAN. Phil. Mag., 21, 535-53.—A mathematical analysis of the relation of heat of mixt. of related substances on the basis of the assumptions; (1) that, when 2 substances are mixed and no new mols. are formed, the heat of mixt. is the change in the total potential energy due to the chem. attraction between the mols.; (2) that the attraction between mols. follows the inverse fifth power law (cf. preceding abstr.).

ROBT. F. EARHART.

Relation between the Three Triple Points of Sulfur. H. R. KRUYT. van't Hoff Lab. Utrecht. Chem. Weekblad, 8, 743-8.—Van't Hoff in 1901 showed that the relation between the temps. of the 2 triple points of S is  $Q_1/T_1 - Q_r/T_r + Q_m/T_m = O$ , where  $Q_r$  and  $Q_m$  are the heats of melting of rhombic and monoclinic S, and  $Q_1$  the heat of transformation of the former into the latter;  $T_r$ ,  $T_m$  and  $T_1$  are the corresponding temps. Insertion of the numerical values confirmed this conclusion, but only through a compensation of errors, as some of the data were quite inaccurate. The agreement is most satisfactory when it is considered as a one-component system, the Q values being calc. from measurements by Brönsted and by Lewis and Randall, the values of  $T_r$ ,  $T_m$  and  $T_1$  being those of Kruyt, Smith and Holmes, and Reicher resp.; but even then it is none too good.

The New Physical Institute of the Stuttgart Technische Hochschule. K. R. Косн. Physik. Z., 12, 818—31.—Description, illustrated with plans and photographs.

J. JOHNSTON.

Remark on a Determination of the Molecular Diameter and the Minimum Optically Effective Film Thickness. J. Koenigsberger and W. J. Müller. Physik. Z., 12, 606; cf. Physik. Z., 6, 847 (1905).—From the mean value for the no. of mols. in 1 cc. of gas at 0° and 760 mm. averaged from the values obtained by Planck, Lorentz, Rutherford, Perrin, Millikan, etc.  $(N = 2.77.10^{19})$  the authors have calc. the mass of 1 sq. cm. of a layer of PbO<sub>2</sub> of mol. thickness. The value thus obtained,  $3.2.10^{-4}$  mg. is about  $\frac{1}{2}$  of the value  $(7.8.10^{-4}$  mg.) given by the authors in their previous paper as the thinnest layer of PbO<sub>2</sub> that can with certainty be detected optically on a Pt mirror. They suggest the possibility that the mol. PbO<sub>2</sub> has the formula Pb<sub>2</sub>O<sub>4</sub> in the solid state, and call attention to the fact that in any case they are able to detect optically a layer not more than 2 mols. thick.

Molecular Flow of Hydrogen through Tubes and the Hot Wire Manometer. MARTIN KNUDSEN. Copenhagen. Ann. Physik, 35, 389-96; C. A., 5, 404.—The flow of H through a very fine capillary tube takes place in accordance with the relations given by the author in previous papers. This indicates that, under the conditions of expt., while the magnitude of the velocity of a gas mol. after impact with the wall is dependent upon the magnitude before impact, the direction of motion after impact is practically independent of the previous direction of motion. Moreover, the expts. show the trustworthiness of the readings of the hot wire manometer (C. A., 5, 1215).

J. JOHNSTON.

Condition of the Atmosphere during the Recent Contact of the Barth with the Tail of Halley's Comet. H. G. A. HARDING. Sydney Techn. Coll. J. Proc. Roy. Soc. N. S. W., 44, 319.—Samples of air, collected during the week previous to, and the week following, the time of contact, were analyzed very carefully and also submitted to spectroscopic exam. The results indicated nothing abnormal in the comp. of the atm.

J. JOHNSTON.

Curious Thermal Phenomenon. E. G. BRYANT. Chem. News, 104, 96, 118.—A bar of metal, coated with a thin layer of wax and red HgI, is heated at one end. After stationary conditions have been attained, the heated end is plunged into cold water, when a slight extension of the zone of transformed HgI will be noticed. J. J.

Compressibility of Mercury. W. C. McC. Lewis. Chem. News, 104, 115.—Abstract of paper read before the British Association. In the case of Hg there is a great discrepancy between the observed latent heat of vaporization and that calc. from the equation proposed by L. (C. A., 5, 3362) using recent data for  $\alpha$ ,  $\beta$  and  $\rho$ . This divergence is ascribed to error in Bridgman's detn. of  $\beta$ , (C. A., 3, 1238); some possible sources of error in Bridgman's experimental work are indicated, all of which would tend to give too high values of  $\beta$ .

J. JOHNSTON.

The Influence of Solvents on the Optical Rotation of Optically Active Compounds.

B. Landau. Chem. Zig., 34, 1032.—A review.

E. J. WITZEMANN.

Composition and Vapor Tension of Solutions. III. Influence of Temperature on the Composition of the Vapors of Solutions. M. S. VREVSKII. J. Russ. Phys. Chem. Soc., 42, 702-14.—The proportion of H<sub>2</sub>O in the vapor of solns. of MeOH and EtOH increases with the temp., while in the case of C<sub>2</sub>H<sub>2</sub>OH it diminishes. Curves are given showing the relation of the components in the vapor at various temps. The comp. of the constant b. soln. of C<sub>2</sub>H<sub>2</sub>OH is deduced graphically. H. M. GORDIN.

Molecular Structure and Optics of Large Fluid Crystals. O. Lehmann. Karlsruhe.

Ann. Physik, 35, 193-219; Physik. Z., 12, 540-6; Arch. sci. phys. nat., 32, 5-19.—

A detailed account of observations with fluid crystals, which cannot be briefly abstracted.

J. Johnston.

Conical Structure Changes in Fluid Crystals. O. LEHMANN. Ber. physik. Ges., 1911, 338-44. J. J.

Influence of a Magnetic Field on Fluid Crystallin Substances. Gerhard Vieth. Halle a/S. Physik. Z., 12, 546-7.—V. observed that the rate of growth of the first fluid cryst. phase of the Et esters of ethoxybenzalamino-α-methylcinnamic acid and of the analogous ethylcinnamic acid increases under the influence of a magnetic field.

I. JOHNSTON.

Viscosity of Fluid Crystallin Mixtures of p-Azoxyanisole and p-Azoxyanetole. HANS PICK. Techn., Hochschule, Breslau. Z. physik. Chem., 77, 577-86.—On the basis of the analogy between flow-pressure of solids and viscosity of liquids, and of the fact that the flow-pressure of mix-crystals is in all the cases investigated much

greater than that of either of the components (Kurnakov and Zhemchuzhnuii, C. A., 3, 303), P. investigated the viscosity of mixts. of p-azoxyanisole and p-azoxyphentole, but found no max. On the contrary the curve is slightly concave, the values diverging little from those calc. from the simple mixt. law. The d. at 136° follows the mixt. law.

J. JOHNSTON.

The Densities of Coexistent Phases and the Diameter of Sulfur Dioxide near the Critical Point. E. Cardoso. Compt. rend., 153, 257-9.—C. finds that in the vicinity of the critical point the line drawn through the points of mean d. shows a pronounced inflection in the case of SO<sub>2</sub>. Corresponding to the crit. temp. there is an ordinate 0.513 which defines the crit. d. Extrapolation of the rectilinear diameter would give a value of 0.520. C. considers that the most probable explanation of this behavior is to be found in the retardation of vaporization.

J. Hunt Wilson.

A General Relation between Heat of Vaporization, Vapor Pressure, and Temperature. I. W. Cederberg. Z. physik. Chem., 77, 498-509.—By combining the equation of van der Waals,  $\log \pi_0/p = a(\theta_0/T - 1)$ , with the Clausius-Clapeyron equation,  $\lambda = T(V - v)dp/dT$  and the Nernst equation,  $p(V - v) = RT(1 - p/\pi_0)$ , C. obtains the expression  $\lambda = R\theta_0 \log [\pi_0(1 - p/\pi_0)/p]/M(\theta_0/T - 1)$ , which is a new relation between  $\lambda$ , p and T, containing no empirical const. This equation holds generally for temps. in the vicinity of the b. p. and makes it possible to calc. the heat of vaporization from the critical consts. The const. a in the above equation of van der Waals can be calc. from the critical and thermal data.

J. Hunt Wilson.

The Cause of Color Production in Inorganic Compounds and their Elements. C. REICHARD. *Pharm. Zentr.*, 52, 591-7.—Illustrations are given to show that the color of salts is not dependent on the particular components. Variation of color may accompany chem. change, change of temp., valence, proportion of H<sub>2</sub>O, and degree of subdivision.

Anton Voriser.

The Color of Iodine Solutions. H. Ley and K. v. Engelhardt. Chem. Inst., Univ. Leipzig. Z. anorg. Chem., 72, 55-62.—The authors repeat previous work (C. A., 5, 282) on the absorption spectra of I solns., the results of which did not agree with those of Waentig (C. A., 4, 854) or of Crymble, Steward and Wright (C. A., 4, 2469). The absorption spectra of I solns. in EtOH change rapidly with the age of the soln., a soln. 22 days old showing a max. at  $I/\lambda = 2850$ , while the fresh soln. showed a minimum. In a study of the effect of solvent no such significant difference as that found by Waentig (C. A., 4, 84) was found by the authors. The question of the constitution of the violet I soln. is not yet definitly settled.

George W. Morey.

Use of Indicators for Determining Affinity Values. V. H. Veley. Chem. News, 104, 115.—Abstract of paper read before the British Association. A brief account was given of the use of indicators for detg. affinity values, with a detailed discussion of the advantage, limitations and defects of the method. The numerical results obtained are independent of the more or less conflicting theories of indicators.

J. JOHNSTON.

Chemistry of the Silver Voltameter. A. S McDaniel. Science, 34, 159-60.—
The relative advantages of the various forms of diaphragms recommended for use in the Ag voltameter are considered briefly. Ordinary filter paper is covered with a very thin layer of oxycellulose; this can be extracted with water, but forms again when the paper is exposed to air. Solns. of oxycellulose reduce Ag solns. with sepn. of colloidal Ag. The reducing action is probably due to the presence of furfuraldehyde, formed by the decomp. of the oxycellulose. The increase in wt. of the Ag deposit is probably caused by the deposition of the colloidal Ag, and may be augmented by small quantities of the reducing body. This would also account for the absence of cryst.

form in the deposit. With a silk disphragm the same behavior is observed, although when used repeatedly the deposits decrease in wt., owing probably to the formation of amino acids. Porous pots when properly prepared are practically inert. It is important that the AgNO, be neutral. The formation of reducing substances, which have been reported as occurring at the anode, is ascribed to the filtration of the anode liquid through paper.

J. Hunt Wilson.

Relation between Color and Constitution of Organic Compounds. A. E. PORAI-KOSHITZ. St. Petersb. J. Russ. Phys. Chem. Soc., 42, 1237-79.—The assumption that color is due to chromophore, auxochrome and quinone groups is not sufficient to cover all cases. In analogy with the explanation of the selective absorption of desmotropic substances, and with Baeyer's theory of the color of fuchsin dyes (Ann., 354, 152), the author ascribes color to intramolecular oscillations, and elucidates his theory on a large number of colored organic compds.

H. M. GORDIN.

Electrical Conductivity in Aniline, Methylaniline and Dimethylaniline. A. SAKHANOV. Agr. Inst., Moscow. J. Russ. Phys. Chem. Soc., 42, 683-90.—A detn. of the conds. of solns. of NH<sub>4</sub>I, LiI, AgNO<sub>2</sub>, Py.HBr and PhNH<sub>4</sub>.HBr in above solvents at 25° showed that the mol. conds. of these substances increase with dilution. This may be due to the formation of conducting complexes of solvent and solute (cf. Z. physik. Chem., 55, 179). In equal dilution the conds. in PhNH<sub>2</sub> were the greatest, next came PhNHMe, and then PhNHMe<sub>2</sub>.

H. M. GORDIN.

Differences of Potential between Cadmium and Alcoholic Solutions of some of its Salts. F. H. GETMAN. Am. Chem. J., 46, 117-30—Measurements have been made of the potential of a Cd electrode in solns. of CdCl<sub>2</sub> and CdI<sub>2</sub> in H<sub>2</sub>O, MeOH and RtOH of varying concs. In aq. solns. the potential of the Cd becomes less negative with increasing conc.; while with the alc. solns. the opposit behavior is observed. For example in the case of CdI<sub>2</sub> in MeOH the values as measured against the calomel electrode ranged from —82.6 millivolts at a conc. of 0.01 M to —121.0 at 0.20 M. In water the corresponding values were —192.3 at 0.01 and —180.1 at 0.20. Slight irregularities were found with CdI<sub>2</sub> in MeOH. The magnitude of the soln. pressures of Cd in aq. solns. of CdCl<sub>2</sub> and CdI<sub>3</sub> have been calc., using the values for the degree of ionization obtained from cond. and f. p. measurements. The values, while uncertain, are of the same order of magnitude as those given previously by Neumann. Since no data are available at present for calcg. the degree of ionization of the alc. solns. of the Cd salts it is impossible to give values for the soln. tension in these solvents.

J. Hunt Wilson.

The System Water-Calcium Chloride-Calcium Hydroxide at 25°. F. A. H. Schreinemakers and T. Fighe. Chem. Weehblad, 8, 683-8.—The authors have investigated the above system at 25°, analyzing both the liquid and the solid phase in about 20 different mixts. The results, which are presented in full and discussed, demonstrate the existence within certain definit conc. limits of the solid double salts CaO.CaCl<sub>2</sub>.2H<sub>2</sub>O and 4CaO.CaCl<sub>2</sub>.14H<sub>3</sub>O.

J. JOHNSTON.

Influence of the Solvent on the Equilibrium Constant. L. PISARZHEVSKII AND I. LITVIN. Inorg. Lab. Polytech., Inst. Kiev. J. Russ. Phys. Chem. Soc., 42, 1062-8; cf. C. A., 5, 610.—An exam. of the values of the equil. const. in aq. Me<sub>2</sub>CO, MeOH and EtOH for the reaction 2AgCN + KBr KAg(CN)<sub>8</sub> + AgBr, and in aq. soln. of mannitol for the reaction, AgCl + KCNS AgCNS + KCl, showed that there is no direct relation between the value of the const. and the viscosity of the solvent or changes in the free energy.

H. M. GORDIN.

Equilibrium of Two Mutually Convertible Substances in a Mixed Binary Solvent. VII. System,  $BaSO_4 + H_2O_4 + H_2O_6 +$ 

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Russ. Phys. Chem. Soc., 42, 1180-94.—Curves and tables are given showing the relation between the equil. of the different phases and the temps. of transformation of the substances in the equation,  $BaSO_4 + H_2SO_4 + H_2O_4 + H_2O_5 + H_2O_6 + H$ 

H. M. GORDIN.

Solutions from the Standpoint of the General Principles of Dynamics. E. Volk-Honskii. Petrovo-Rasumovskoe, Moscow. J. Russ. Phys. Chem. Soc., 42, 1194-7.—Mathematical discussion of applications of D'Alembert's principle (cf. Mach, Mechanics) to solns.

H. M. GORDIN.

Combined Solubilities in Water of Carbonate and Bicarbonate of Sodium. Generalization of a Formula of van't Hoff. EDOUARD HERZEN. Bull. soc. chim. belg., 25, 227-34.—A criticism of a claim by de Paepe (C. A., 5, 2604) that Na<sub>2</sub>CO<sub>2</sub> and NaHCO<sub>3</sub> in conc. aq. soln. are resp. ionized according to the scheme: Na<sub>2</sub>CO<sub>2</sub> = Na<sup>+</sup> + NaCO<sub>2</sub><sup>-</sup> and NaHCO<sub>3</sub> = H<sup>+</sup> + NaCO<sub>2</sub><sup>-</sup>. H. adapts the van't Hoff formula for combined solubilities of substances having a common ion to the case where the degrees of dissociation of the component substances are different; assuming NaHCO<sub>3</sub> = Na<sup>+</sup> + HCO<sub>3</sub><sup>-</sup> for the ionization of NaHCO<sub>3</sub> he finds, by the use of the generalized formula, that the experimental data for the combined solubility of Na<sub>2</sub>CO<sub>3</sub> and NaHCO<sub>3</sub> accord equally well with the schemes Na<sub>2</sub>CO<sub>3</sub> = 2Na<sup>+</sup> + CO<sub>3</sub><sup>-</sup> and Na<sub>2</sub>CO<sub>3</sub> = Na<sup>+</sup> + NaCO<sub>3</sub><sup>-</sup>. It is pointed out that de Paepe, in assuming the scheme NaHCO<sub>3</sub> = H<sup>+</sup> + NaCO<sub>3</sub><sup>-</sup>, has implied the existence of a considerable conc. of H ions in an alk. medium.

H. S. PAINE.

The Dissociation Pressures of Alkali Bicarbonates. I. Sodium Hydrogen Carbonate. R. M. CAVEN AND H. J. S. SAND. J. Chem. Soc., 99, 1359-69; Proc., 27, 147.—The authors have investigated the equil. represented by the equation 2NaHCO<sub>3</sub> = Na<sub>3</sub>CO<sub>3</sub> + H<sub>2</sub>O + CO<sub>2</sub> by measuring the vapor pressures produced on heating mixts. of NaHCO<sub>2</sub> and anhydrous Na<sub>2</sub>CO<sub>2</sub>. Measurements of the vapor pressure of Na<sub>2</sub>CO<sub>2</sub>.H<sub>2</sub>O showed that this substance does not exist in the mixt. under the conditions of the expts., since its vapor pressure is lower than the equil. pressure, corresponding to the above equation. Mixts. of NaHCO, and Na, CO, were heated in an evacuated flask and the vapor pressure generated was balanced against air, the pressure being measured by means of a sensitive manometer. In order to ensure a gaseous phase of equimol. proportions of CO, and H<sub>2</sub>O, large quantities of gas were drawn off from the mixt. at high temp. until successive readings were found to be identical. Trouble was experienced, particularly at temps. below 95°, owing to the formation of crusts on the mixt. of salts. This caused a marked retardation in the establishment of equil. By using large amts. of the mixt. and heating rapidly this difficulty was overcome. The vapor pressure of Na<sub>2</sub>CO<sub>2</sub>.H<sub>2</sub>O was measured at temps. ranging from 17.5° to 97.7° and that of NaHCO<sub>2</sub> from about 20° to 110°. The vapor pressure of Na<sub>2</sub>CO<sub>2</sub>.H<sub>2</sub>O can be reproduced with fair accuracy by the expression  $\log p = 10.824 - 3000/T$ , and that of NaHCO<sub>3</sub> by a similar equation,  $\log p = 11.8185 - 3340/T$ . The heats of dissociation have been calc. to be 13,800 and 30,700 cals., resp. Using the values obtained for the vapor pressure of Na<sub>2</sub>CO<sub>2</sub>.H<sub>2</sub>O and NaHCO<sub>2</sub> and applying the law of mass action, it is possible to calc. the equil. pressure for the four-phase system Na<sub>2</sub>CO<sub>2</sub>, NaHCO<sub>2</sub>, Na<sub>2</sub>CO<sub>2</sub>, H<sub>2</sub>O, and vapor. Within the range of temps, covered these values are a little higher than the vapor pressure of NaHCO, J. HUNT WILSON.

Application of the Phase Rule to Mineral Silicates. V. M. GOLDSCHMIDT. Christiania. Z. Elektrochem., 17, 686-9.—G. states a specialized form of the phase rule, especially applicable to mineralogy, as follows: The max. no. n of minerals which, at a given pressure and temp., other than the transition pt., can exist in contact with their satd. soln. is equal to the no. n of components into which the minerals may be split up. He says this rule is applicable to those systems in which one solid mineral changes

into another without passing through a homogeneous state, e. g., contact metamorphosis. In contact products where only MgO, SiO<sub>2</sub>, and Al<sub>2</sub>O<sub>3</sub> are present, only 3 different minerals can coëxist. When CaO is present, there may be 4. G. has investigated the contact minerals of the Christiania district and has never found more coëxisting minerals than could be accounted for by the phase rule.

J. L. CRENSHAW.

Simultaneous Autocatalysis and Negative Catalysis. A. QUARTAROLI. Ist. tecn. Viterbo. Gass. chim. ital., 41, II, 64-9.—In the formation of N<sub>2</sub>O from KNO<sub>2</sub> and HCO<sub>2</sub>H, N<sub>2</sub>O<sub>3</sub> is first formed, this then reacting with the acid. The second reaction is rapid only at 40°; if the mixt., instead of being heated suddenly, is kept at 20°, the liquid becomes intensely blue. The reaction begins very slowly, then becomes tumultuous and finally slows down, indicating autocatalysis. The positive catalyst, according to the author, is the N<sub>2</sub>O<sub>3</sub>, and if some substances were present which would at once oxidize the HNO<sub>2</sub> to HNO<sub>3</sub>, the reaction should stop, or at least be slowed down. Expts. with HClO<sub>4</sub>, H<sub>2</sub>O<sub>2</sub>, KMnO<sub>4</sub> and urea showed that such was the case.

Chas. A. ROUILLER.

Diffusion Experiments. O. SCARPA. Napoli. Gasz. chim. ital., 41, I, 113-21, 122-6; cf. Nuovo cim., Sept., 1910.—Some of the causes of the discrepancies between the true diffusion coeff. and the values found by applying the theory of diffusion to Vanzetti's expts. (C. A., 2, 2898, and J. Chem. Soc., 1909, II, 978) are discussed. The chief discrepancies are due to phenomena of false equil. In expts, with HCl and AgNO<sub>2</sub>, the whole of the discrepancies may be accounted for by assuming the max. possible value of supersatn. of the AgCl at the moment of formation, yet in the case of NaCl and KCl, the correction thus obtained, while of the proper sign, is not nearly sufficiently large, and the discrepancy is probably due to the AgCl assuming the colloidal form before pptg. in the tube, an assumption strengthened by the conditions under which the ppt. is formed and its appearance when it first becomes visible. zetti's expts. with HCl, NaCl and KCl were repeated and while with capillaries which had been previously washed with KCN to remove every trace of AgCl anomalous results similar to Vanzetti's were obtained, if the tubes were merely washed with H.O and used several times so that some of the ppt. remained on the walls, the conditions thus being made unfavorable for false equil., the results were found to agree quite closely with those calc. from the theory. The point at which the ppt. first began to form, detd. by means of a lens and lateral illumination (Tyndall), not the point at which the diaphragm was formed, was, in every case, taken. CHAS. A. ROUILLER.

Diffusion Experiments. B. L. Vanzetti. Milano. Nuovo cim., 20, II, 442-4.

—Answer to Scarpa (preceding abstr.).

C. A. R.

Photokinetics of Bromine Substitution. I. Course of the Photo-reaction. L. BRUNER AND S. CZARNECKI. II. Chem. Univ. Lab., Cracow. Bull. intern. acad. sci. Cracovie, 1910, 516-59; cf. C. A., 5, 3045.—Br substitution in toluene was first studied, using Nernst lamps of 25 and 80 c. p. as source of light. Even traces of O have a disturbing influence on the course of the reaction. Efforts to free the solns. and app. from O were unsuccessful, but it was found that slight amts. (0.0004-0.003 mols.) of I made the reaction independent of the presence of O. The retarding and subsequent accelerating action of O may probably be explained by the formation of a labil Br-O compd. in which I may displace and liberate Br. The reaction rate is independent of Br conc. and equal amts. of Br are consumed in equal time intervals. The reaction velocity is proportional to the toluene conc. and decreases with increasing I conc. Starting with an expression for the reaction rate which takes into account the coeff. of transparence of the soln., the thickness of liquid layer exposed to light, and the light intensity, and making some simplifying assumptions, it is found that n, the order of the reaction, is I (as was previously found for the same reaction when taking place

in the dark). The temp. coeff. between 1° and 33° is 1.85 (about ½ as great as for side-chain substitution in the dark). Expts. in which different solvents were used showed that the difference in reaction velocities for ionizing and indifferent solvents becomes less as greater light intensities were used. The relative light susceptibility of substitution products and homologs of toluene may be arranged as follows in the order of increasing sensitiveness: toluene, m-xylene, o-xylene, p-xylene, Et benzene, isopropylbenzene, and cymene. The effect of different wave lengths on photo-bromination was studied, using a Hg lamp; very probably the wave length of light has no sp. influence on the reaction rate, aside from the energy involved. H. S. Pains.

Adsorption Experiments. K. Scheringa. Chem. Weekblad, 8, 11-2.—S. finds that the conc. of Pb in aq. solns. of Pb salts is diminished by contact with paraffin wax, and ascribes this to soln. in the wax.

J. JOHNSTON.

Negative Adsorption. R. O. Herzoc. Z. Chem. Ind. Kolloide, 8, 209-10.—H. claims that Neuner (C. A., 5, 2444) misrepresents him in quoting from his work on negative adsorption (C. A., 5, 1539). The explanation of negative adsorption as being due to a semipermeable membrane about the colloid was suggested because of its simplicity. H. admits that in cases where the colloid material is not adsorbed by the gel the phenomenon may equally well be explained as an adsorption of water by the gel. Emulsion colloids are most apt to show negative adsorption. H. ISHAM.

Viscosity and Fluidity of Suspensions of Finely Divided Solids in Liquids. EUGENE C. BINGHAM AND T. C. DURHAM. Richmond Coll., Va. Am. Chem. J., 46, 278-97.-Measurements were made, at a series of temps., of the fluidity of suspensions of infusorial earth, china clay, and graphite ("aquadag") in H<sub>2</sub>O and of infusorial earth in EtOH; the capillary tube method was used. The fluidity decreases rapidly and linearly as the vol. conc. of the solid increases. At a definit conc., which is independent of the temp, and of the particular app, employed, a zero fluidity is reached, which apparently demarcates viscous from plastic flow. This comp. is not the same for different substances suspended in the same liquid, nor is it necessarily the same for one substance suspended in different liquids, Small amts, of impurities change the character, and alter the fluidity, of the suspensions. Comparative expts. on various pure liquids with the Ostwald viscometer showed that this app. may yield inaccurate results, unless the rate of transpiration is great; the inaccuracy is due to the fact that, as ordinarily used, it fails to take into consideration the losses of kinetic energy which are variable with the temp. and from one liquid to another. J. JOHNSTON.

Polychroism of Silver. LOPPO-CRAMER, van Bemmelen Gedenkboek, 143-6.— Evidence is adduced in favor of the view that the color of metal suspensions varies with the size of the metal particle. In the case of fine suspensions of Ag in gelatin, the color changes from yellow through red to blue as the size of particle increases.

J. JOHNSTON.

Structure of Gelatinous Silicic Acid. Theory of Dehydration. RICHARD ZSIG-MONDY. Inst. anorg. Chem. Göttingen. Z. anorg. Chem., 71, 356-77.—O. Bütschli, in 1900, on the basis of careful microscopic investigations, concluded that the dry gel of SiO<sub>2</sub> contains empty spaces of microscopic dimensions. Z. has now made ultramicroscopic observations which show that the structure is much finer meshed than Bütschli imagined it to be. The structure is mainly amicroscopic; the small hollow spaces are all interconnected, since a rapid and complete permeation of the gel by various liquids (H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, etc.) is possible. Z. presents the view that the gel is made up of very fine grains sepd. by what are in effect very fine capillary tubes, and shows how this view harmonizes with the phenomena observed on dehydrating and rehydrating SiO<sub>2</sub> gels. For instance, the diminution of vapor tension of the water is a direct

consequence of the fineness of the capillary. Assuming the validity of the laws of capillarity for very small capillaries, and a vapor pressure lowering of 6 mm., the author calcs. the average diam. of the spaces between the particles of a SiO<sub>2</sub> gel to be about 5µµ. The optical behavior of the gel, and its applicability for ultrafiltration, support the view that the dimensions are of this magnitude. The phenomena observed on dehydrating, re-hydrating, and heating SiO<sub>2</sub> gels are discussed in detail with special reference to the point of view here advocated.

J. JOHNSTON.

The Capillary Analysis of Colloidal Solutions. F. Fichter and N. Sahlbom. Basel. Verk. Naturf. Ges. in Basel, 21, 1-24; Chem. Zentr., 1910, II, 1088.—In capillary analysis the + colloids are pptd, when brought in contact with filter paper. With thick papers which had not been washed with acids the test could be made in the open air; with thinner papers only when evapn. was prevented. The nature of the paper causes variations in the results. Negative colloids rise with the water without any pptn. The pptn. is similar to that in capillary tubes, which is a result of the e.m. f. produced by the rise in the capillary. This causes the walls of the capillary to become negatively charged and the + colloid to ppt. thereon. The pptn. tension for Graham's Fe oxide sol. decreases rapidly with the conc. of the sol. The tension, calc. from the critical diam. of the capillary (0.15 mm., the largest diam. at which pptn. occurs), is about 0.67 v. The incomplete pptn. of partially dialyzed solns. of a + colloid is to be explained by the influence of the free acid, which raises the pptn. tension or peptinizes the ppt. More or less hydrolyzed salt solns. are broken up by the capillary strips into the undecomposed salt and the colloidal hydroxide which ppts. These phenomena may be used in investigations of the process of dialysis of + colloids, in investigations of paper and of the degree of hydrolysis of salt solns.

Capillary Analysis of Colloidal Solutions. N. Sahlbom. Basel. Kolloid Chem. Beihefte, 2, 79-140.—See preceding abstr. H. I.

The Capillary Electric Precipitation of Positive Colloids. F. FICHTER. Z. Chem. Ind. Kolloide, 8, 1-2.—Positively charged colloids are pptd. by filter paper or by capillary tubes below the "critical" diam. (0.15 mm.) (see preceding abstrs.). The same phenomenon was found to occur when a drop of + sol was placed on a clean glass plate and another plate lowered upon the first. When the proper distance was reached a ring of ppt. appeared, which grew inward as the plates came nearer together. The critical distance for the plates should be 0.075 mm., but was always found to be less (about 0.03 mm.), owing to a transfer outward of the first ppt. as the drop is pressed still further out. The experiment is thus only of qualitative interest, but offers an explanation of certain phenomena formerly considered adsorption effects; e. g., when a mixture of acid and basic dye is dropped on filter paper, the basic (+) remains pptd. on the paper where it first touches while the acid (—) spreads out with the water.

H. ISHAM.

The Micellar or Colloidal Condition. G. Malfitano. Kolloid. Chem. Beihefte, 2, 142-212.—A general review of the subject followed by an enlargement of a previous article (C. A., 4, 706). The colloidal condition is defined as that which occurs when insol. mols. are formed in a liquid under such conditions that they can unite with other mols. capable of electrolytic dissociation, and the colloid is most stable when I element or radical is common to both mols. The compd. can be expressed by the association of a variable no. of insol. mols. M with easily ionized mols.,  $A \pm B \mp$ , as  $(M^*A \pm B \mp)$ . The first steps in this process, when n is small, may result in crystalloids as in the case of  $(HgS)_1(H_2O)_1K_2S$ , while an increase in n gives a colloid. A may be a polyvalent ion; if B is monovalent a sol results, if polyvalent pptn. occurs. H. ISHAM.

Simultaneous Congulation of Two Colioids. F. W. TIEBACKX Z. Chem. Ind.

Kolloide, 8, 198-201.—A mixt. of 2 cc. 0.5% gelatin and 2 cc. 2% gum arabic solns. is coagulated by addition of 2 cc. 0.025-0.01 N HCl or  $H_2SO_4$ ; for other acids the range in conc. for coagulation varies considerably. More conc. or more dil. acids give a clear soln. The coagulum may be removed by filtration, is insol. in hot water but readily sol. in acids. The amt. of acid necessary for coagulation increases with the amt. of gum present, and the coagulation is most complete when gelatin and gum are present in about equal quantities, and when the acid is added rapidly; changes in the gelatin conc. have little effect. The acid is adsorbed by the gum, thus charging it positively; if the unadsorbed acid has reached a conc. of 0.01 N the gelatin also becomes positive. Between these limits lies the coagulation zone. The coagulum is an adsorption compd. which swells in salt solns., the anions and cations being effective in the following order:  $I > Br > Cl > SO_4 > CO_4$ , K > NA > alk. earths I > SI > Mg. Salts of the weaker acids interfere with coagulation, those of the stronger acids have the same effect to a less degree.

Emulsion Colloids and Observations on the Methods of Determining Ultramicroscopic Particles. G. Wiegner. Göttingen. Kolloid. Chem. Beihelte, 2, 213-42; Chem. Zentr., 1911, I, 1397.—The name emulsion or suspension should be applied to such systems as spontaneously sep.; true emulsions, which do not sep. spontaneously, should be designated as emulsion colloids or emulsoids. Systems of low degree of dispersion which do not sep. by the addition of chem. agents are pseudoemulsions. Emulsions of olive oil and water were prepared by a Laval emulsifier, so that there was no sepn. under the influence of gravity. The no. of oil particles as detd. by the Siedentopf-Zsigmondy ultramicroscope was 5 × 10° per cc. Such emulsions show the typical colloid properties of negative sols., such as hysteresis, coagulation by electrolytes, colloidating action and pptn. by other colloids. The degree of dispersion at which sedimentation no longer occurs, and when truly colloidal properties are acquired, depends on all the factors which det. the Brownian movement and may be different for different emulsions and for different conditions of the same emulsion.

H. ISHAM.

Coagulation of Colloids. V. A. VOLZHIN. J. Russ. Phys. Chem. Soc., 42, 863-76.—Two solns. of Prussian blue were prepared by adding FeCl<sub>2</sub> to a soln. of K<sub>4</sub>Fe(CN)<sub>6</sub>. (a) by using excess of FeCl<sub>2</sub>, washing the ppt. and dissolving it in (CO<sub>2</sub>H)<sub>2</sub>; (b) by using excess of K<sub>4</sub>Fe(CN)<sub>6</sub> in sufficient amt. to prevent pptn. To these solns. in different dilns. varying amts. of NaCl, NH<sub>4</sub>Cl and KCl were added, and the quantities required to produce coagulation represented by curves. The results show that the comp. of the gel varies with the conc. of the soln. A comparison of the relative amts. of NaCl, NH<sub>4</sub>Cl and KCl required for coagulation showed that these were as 4:2:1 for (a) and as 4:4/3:1 for (b).

Ultraviolet Absorption Spectra of the Vapors of Various Organic Substances Compared with the Absorption of these Substances in Solution and in Thin Films. J. E. Purvis. Chem. News, 104, 115-6.—Abstract of paper read before the British Association. The results are discussed from the point of view that the movements of the atoms are influenced by their nature, wt., type and orientation. The vapor mols. have considerable freedom of movement, and a considerable number of bands are in general produced. In soln. the solvent acts partly as a constraint on the vibrations and on the number of encounters, and partly as an absorbent of the radiant energy, so that the narrow bands of the vapors are usually replaced by wide diffuse bands. In very thin films the movement of the mols. is still further restricted, but the selective absorption is not unlike that of the solns., the chief difference being that the bands of the thin films are shifted more towards the less refrangible regions.

J. JOHNSTON.

Absorption and Dispersion in Metallic Vapors. P. V. BRVAN. Chem. News, 104, 116-7.—Paper read before the British Association. A general discussion of the subject.

J. JOHNSTON.

Another Case of Ionization of Gases by Chemical means. Angel Del Campo y Cerdan and Jame Ferrer. Univ. Central. Rev. real. acad. cien., Madrid, 9, 451—62.—The phosphorescence (first observed by Delépine) of dimethylsulfothiocarbonate is believed to be a case of triboluminescence influenced by pressure; the chem. action (which is anterior to production of the white fumes) is probably saponification followed by oxidation (hence dependent on the presence of moisture and O). Ionization of the air accompanies this phenomenon, as shown by the rate of discharge of a Curie electroscope; only slight evidence could be obtained of ultraviolet radiations or of radiations capable of impressing a photographic plate through an opaque body (see C. A., 5, 3682).

Some Peculiar Phenomena of Photoluminescent Substances. José Rodriguez MOURELO. Madrid. Rev. real. acad. cien., Madrid, 8, 737-55.—Discussion of the results of M's work on the subject. He regards photoluminescence as a phenomenon of mol. dissociation in dil. solid solns. simultaneous with a reversible chem. reaction between diluent and active substance. CaS, BaS, and SrS were used as diluents and nitrates of Bi, Mn, UO, Cu, Ni, Co and chlorides of Sb and Th (sometimes accompanied by borax, NaCl, Na<sub>2</sub>CO<sub>2</sub>, or K<sub>2</sub>SO<sub>4</sub>) were used as active substances. In order to secure proper homogeneity the sulfides of Ca, Ba, and Sr should be formed in the mass from the appropriate carbonates and flowers of S by heating; an optimum range of temp. for accomplishing this was observed, the sensitiveness to light of the resulting mass being impaired by too high or too low a temp. Ternary, quaternary and more complex systems (regardless of whether the diluents exceed the active substances in no. or vice versa) are less photoluminescent than binary systems and behave as though composed of various possible binary systems whose photoluminescent activities are more or less mutually neutralized, the sensitiveness to light and the color and intensity of the luminescence being that characteristic of the binary system which is most active when isolated (this is modified in some degree, however, by the quant. relations).

Spectra of Agron. W. STAHL. Z. wiss. Phot., 9, 302-12.—S. studies the vacuum tube spectrum of A under 3 conditions: (1) At a pressure of 3 mm. (red discharge); (2) at a pressure less than 3 mm. with capacity in parallel (blue discharge); (3) at a pressure of 30 mm. with strong current and large capacity in parallel. The object of the investigation is to det. whether or not there is any spectroscopic evidence tending to show that A is not a single gas. The results are negative. Several tables of wave lengths are given. Although some of the lines of the "white" spectrum are considerably broadened toward the red, S., employing a much greater dispersion, fails to confirm the measurements of Eder and Valenta who, in some cases, record a shift of 0.5-1.0 Å. u. toward the red.

The Long-wave End of the Arc and Spark Spectrum of Copper. M. Aretz. Z. wiss. Phot., 9, 256-69.—Previous investigations on the spectrum of Cu were carried out at a time when the sensitiveness of photographic plates extended only to about  $\lambda=6000$  Å. u. The present investigation was made possible by the introduction of Wratten and Wainwright plates whose range of sensibility extends in the red even beyond  $\lambda=750~\mu\mu$ . A 6 in. Rowland concave grating of 6.6 m. radius of curvature and 16,000 lines per in. served as the dispersive app. The dispersion was such that, on the photographs, 1 mm. covered 2.4 Å. u. All measurements of wave length are referred to the Fabry and Buisson Fe standards. The range of wave lengths covered in the study of the spark spectrum extends from  $\lambda=4700-\lambda=5782$  Å. u.

while in the case of the arc spectrum the range extends from  $\lambda = 5034-\lambda = 6920$  Å. u. It is noted that the lines of the Cu spark and arc in air are, as a rule, hazy and broad and the marked difference between the arc and spark spectra is discussed. A complete table of wave lengths is given in the original.

A. H. Pfund.

Photometry of Lights of Different Color. M. v. Pirani. Z. wiss. Phot., 9, 270-2. -A comparison of the intensities of illuminants which differ markedly in color is accompanied by great difficulties. In order to make such a comparison possible P. employs color screens whose max. of transmission lies at the wave length 550 µµ which likewise is the wave length to which the human eye is most sensitive (according to measurements by König). Such a color screen, in being placed between the eye and the photometer, gives very nearly the same yellowish green color to the 2 sources whose intensities are to be compared—thus making possible an accurate detn. The 2 (superimposed) color screens employed had the following transmission coeffs. Gelbglas No. 5899. Schott & Gen. Thickness 4 mm. 644 μμ, 0.89; 578, 0.96; 546, 0.95; 509, 0.81; 480, 0.008. Zettnow filter. Zeiss. Thickness 1.9 mm. 644 μμ, 0.25; 578, 0.43; 546, 0.55; 509, 0.31; 480, 0.08. With this arrangement P. compares an Hg vapor lamp, a W lamp burning at o.6 watts per candle and a C incandescent lamp, burning at 3.5 watts per candle. The max. difference in the measurements of 3 observers amounted to ±0.5% while a direct comparison of intensities yielded a difference of ±1.6%. It is suggested that all photometers (excluding, of course, flicker photometers) be supplied with a yellowish green screen. A. H. PFUND.

Photo-electric Testers and their Use in Artificial and Atmospheric Fields. H. Dember. Phys. Inst. Tech. Hochschule, Dresden. Ber. K. Sachs. Ges. Wiss. Math.-phys. Kl., 62, 218-50; through Chem. Zentr., 1910, II, 1354.—Bright metal wire under the influence of sunlight assumes in the elec. field of the atm. a potential which approximates to that assumed by a flame collector at a similar height above the earth's surface. The author has investigated the mode of action and the reliability of this photo-elec. potential equalizer. Under the influence of the light, electrons leave the metal and are deposited on the gas mols. in the immediate vicinity, leaving a positive charge on the metal. Surrounding the tester there are only negative ions which are dispersed through diffusion and the action of the field. The objection that there is an increased conc. of ions in the immediate neighborhood of such a tester is unfounded. When the final potential is reached, all action outside of the surface of the metal ceases. This form of tester is especially adapted for use in a balloon for detns. of the ion content of the atmosphere.

E. J. Crane.

Coloration of Tinted Minerals (BRAUNS). 8.

Law of Mineral Association (GOLDSCHMIDT). 8.

Platinum Catalysis in the Oxidation of Hydrogen with Sulfuric Acid (MILBAUER).

6.

Melting-point and Deformation Eutectics (STALBY). 19.

Enzyme Studies. II. Hydrogen Ion Concentration in Enzyme Reactions (SÖR-ENSEN). II.

Rate of Hydration of Metaphosphoric Acid (BALAREFF). 6.

Electrical Conductivity of Diamonds (DOELTER). 8.

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### 3. RADIOACTIVITY.

#### HERMAN SCHLUNDT.

Carnotite as a Source of Radium. Anon. Eng. Min. J., 92, 507.—The deposits of carnotite in southwest Colorado and adjoining areas of Utah and New Mexico are now yielding a much more suitable material from which to extract radium. A sample of carnotite found in southwestern Colorado, taken from near the surface was found to possess a radioactivity of 6.2.

ROBERT KANN.

The Solubility of Radium Emanation in Organic Liquids. Eva Ramstedt. Faculté Sciences, Paris. Radium, 8, 253-6.—The method used differed in several particulars from that of previous investigators (cf. Hofmann, Physik. Z., 6, 337 (1905); Kofler, C. A., 2, 1384). Measurements were made at  $+18^{\circ}$ , 0°, and  $-18^{\circ}$ , with glycerol (18° only), water, aniline, alcohol, acetone, ethyl acetate, paraffin oil, benzene, xylene, toluene, CHCl<sub>8</sub>, ether, hexane, cyclohexane, and CS<sub>2</sub>. ( $\alpha$ ) increased from 0.21 for glycerol at 18°, in the order shown, to 23.14 for CS<sub>2</sub> at 18°, and in each case there was a marked increase of ( $\alpha$ ) with decrease of temp. ( $\epsilon$ . g., water 18°, 0.285; 9°, 0.39; 0°, 0.52; CS<sub>2</sub>, 18°, 23.14; 0°, 33.4;  $-18^{\circ}$ , 50.3). The results are in good agreement with those (for much fewer of the solvents) previously found by Hofmann, and Kofler; the arrangement of solvents in order of rise of solubility agrees fairly well with that found by Just for CO<sub>2</sub>, CO, H<sub>2</sub>, and N<sub>2</sub> (Z. physik. Chem., 37, 342 (1901)).

A. T. CAMBRON.

The Ratio between Uranium and Radium in Active Minerals. ELLEN GLEDITSCH. Faculté Sciences, Paris. Radium, 8, 256-73.—The constancy of the U-Ra ratio seemed firmly established by Boltwood's work (Am. J. Sci., 18, No. 104; Phil. Mag., 9, 599 (1905)) together with that of Eve and Strutt. Using different methods, however, results obtained by G. showed marked differences (C. A., 4, 408). Detailed results now given for a large number of U minerals, completely confirm the differences

previously obtained. The figures vary from Ra/Ur = 1.82 × 10<sup>-7</sup> for a Saxe chalcolite to  $3.74 \times 10^{-7}$  for a pitchblende from Cornouailles. Estns. of the Ra and U contents in any one mineral by different methods show close agreement. The greater number of minerals examined show fairly close agreement (limits 3.14 and 3.74  $\times$  10<sup>-7</sup>). The exceptions are chiefly autunites and gummites. Variations for autunite have also been found by Soddy and Pirret (C. A., 5, 827, 828), Russell (C. A., 5, 828), and Marckwald and Russell (C. A., 5, 3007). The variations in particular minerals are due chiefly to weathering produced by running water, while the variations in different minerals are due to the age of the mineral themselves, showing that Ra and U are still in incomplete equil.; this suggests that 35,000 years (the life period for the intermediate product, ionium, calc. by Soddy) must be considered as a minimum value. The method adopted by Soddy to calc. the age of various minerals from the variation in the Ra/Ur ratio, and the He content, is criticized adversely, on account of the variation in He absorption with different minerals. The Ra/Ur ratio varies only within small limits, which can be interpreted by means of the radioactive hypothesis. The relation between the 2 elements, through the intermediate stage ionium, is now well established, and is not affected by the slight inconstancy found. A. T. CAMBRON.

Certain Gas Ionization Effects Observed in the Presence of Non-radioactive Substances. II. Activity and Luminescence of Quinine Sulfate. M. DE BROGLIE AND L. BRIZARD. Radium, 8, 273-9.—The phosphorescence of quinine and cinchonine sulfates during their changes of hydration shows a continually luminous background on which sep. scintillations are visible. Their aspect in rarefied air resembles an elec. discharge. Independently of hydration changes, triboluminescence is shown when these crystals are subjected suddenly to the temp. of liquid air, and at the same time the gas surrounding them is ionized. Absorption expts. show that if this ionization is due to a radiation emitted during luminescence; such radiation does not belong to any of the ultraviolet type yet known. It is suggested that the elec. discharges accompany sudden rupture of the crystals.

A. T. Cameron.

Verification of the Theory of the Brownian Movement, and Determination of the Value of Ne for Gaseous Ionization. H. Fletcher. Phys. Lab., Univ. Chicago. Radium, 8, 279-86.—It is shown that the continual agitation of a small particle suspended in a gas, known as the Brownian movement, is entirely explicable by the kinetic theory of gases, and that the values of Ne obtained for gaseous ions (2.88  $\times$  10<sup>-14</sup>) is the same as that for electrolytes.

A. T. Cameron.

Some Chemical Effects of Radium Rays. S. C. LIND. Faculté Sciences, Paris. Radium, 8, 289-92.—Measurements have been made of the action on HBr, liquid and gaseous, and on its constituent gases, by the penetrating rays from Ra Em., and by the total radiation. The penetrating rays from 200 mg. RaCl. have no perceptible action on gaseous HBr, nor on a mixture of H and Br. The total radiation (when the Em. in equil. with 1 mg. of Ra is mixed with the substance on which it acts) produces a slight combination between hydrogen and bromine; this is much smaller than that observed on H and Cl by the penetrating radiations (Jorissen and Ringer, Ber., 39, 2093 (1906)). The amt. of chem. action produced is apparently not greater at 303° than at ordinary temp. The total radiation from Em. in equil. with 10 mg. Ra liberates in 14 days 0.0328 g. Br from 1.4020 g. liquid HBr. The energy utilized in this expt. is about 3.5% of the total energy liberated by the emanation in the same period. Aq. solns. of HBr are decomposed by the Em. Solns. of KI are decomposed by the penetrating radiations, and in tenfold amt. by the total radiation (cf. Ramsay, C. A., 3, 512). A. T. CAMBRON.

Ionization of Liquid Hydrides of Carbon. T. BRALOBYETZKII. Lab. phys., Univ.

Kiev. Radium, 8, 293-9.—The ionization produced by the penetrating rays from Ra on petroleum ether, ligroin, and petroleum, has been measured. A. T. C.

Attempts to Prepare Metallic Radium. HERSCHFINEEL. Univ. Paris. Radium, 8, 299-301.—H. has repeated Ebler's work (C. A., 5, 628), attempting to obtain metallic Ra by heating Ra-Ba hydrazoate in vacuo (1 mg. Ra in 11 mg. substance). Decomp. took place, with production of small quantities of N, and very impure metal which may have contained a little Ra. It seems likely, that Ra hydrazoate cannot be prepared, since the radiations would decompose it as fast as it was formed.

A. T. C.

The Penetrating Radiation over the Sea. D. PIUTTI. Inst. Cent. Météorolique et Geodyn., Italie. Radium, 8, 307-12.—The number of ions over the sea due to penetrating radiation, is  $^3/_3$  that on land. The number per cc. per sec. is less by 5 than the mean value on land. The penetrating radiations show variations which are at least of the same order of magnitude as those on land. There appears to be no relation between the state of the sea, and the magnitude of  $\gamma$ -radiation, so that the latter cannot be attributed to products of Em. liberated from the sea with more or less facility according to the intensity of surface disturbance. It appears that an appreciable part of the penetrating radiation present in air, especially that which is subject to considerable variation, has an origin independent of the direct action of active substances in the surface layers of the terrestrial crust.

A. T. Cameron.

New Measurements of the Zeeman Effect Shown by Some Emission Bands of Molecules of Substances in the Gaseous State. A. Dufour. Ann. chim. phys., 21, 568-73. —The deviations of the magnetic doublets of the edges of the bands studied (D, D' of CaF<sub>3</sub>;  $\Delta$ ,  $\Delta''$  of CaCl<sub>2</sub>; E, E', E'', of SrF<sub>2</sub>; A, B, C, of BaF<sub>2</sub>; P, of BaCl<sub>2</sub>) increase proportionally to the intensity of the magnetic field. Values for  $\partial \lambda/H\lambda^2$  ( $\lambda$  = length in cm., H = intensity of field, in gauss) are given for the doublets of the different bands. These usually approximate to the normal deviation or bear a simple relation to it.

A. T. Cameron.

Remarks on Marx's Experiments on the Velocity of Röntgen Rays. J. Franck and R. Pohl. Phys. Inst., Univ. Berlin. Ann. Physik, 34, 936-40.—A controversial paper, criticizing Marx's interpretation of his results (*Ibid.*, 33, 1305-91 (1910)).

A. T. CAMERON.

(The Velocity of Röntgen Rays.) Erich Marx. Phys. Inst., Univ. Leipzig. Ann. Physik, 35, 397-400.—A reply to Franck and Pohl (see above). A. T. C.

Experimental Determination of the Variation of Inertia of Cathode Corpuscles as a Function of the Velocity. C. E. Guye and S. Ratnowsky. Arch. sci. phys. nat., 31, 293-311.

H. S.

JACOBS AND GEETS, V.: Le radium. Bruxelles: 8°, 113 pp.

U. S., 1,001,480, Aug. 22. F. ULZER and R. SOMMER, Vienna. Extracting radium compounds by treating a Ra-containing substance, e. g., pitchblende residue, at high temp. (preferably b.) with conc. H<sub>2</sub>SO<sub>4</sub>, washing and filtering the residue, beating the residue with caustic alkali, washing and filtering the resulting residue and treating it with dil. H<sub>2</sub>SO<sub>4</sub> (cf. C. A., 5, 2369).

## 4. ELECTROCHEMISTRY.

C. G. FINK.

The Work of the Physikalisch-Technischen Reichsanstalt in 1910. GRÜNBAUM.



Elektrotechn. Z., 32, 829; Electrician, 67, 818.—The value of the e. m. f. of the Weston cell as detd., at Washington (1.0183 international v.) was adopted. The 4 manganin resistances kept as sec. standards were frequently compared and remained const. An atm. of 50% humidity was kept around the 1-ohm standard during July. The variation was less than 1/100,000th ohm. In searching for the most suitable arrangements for testing transformer oils, spheres 10 and 15 mm. in diam. as well as points were used. With dry, clean oil the breakdown between the points was a little lower than between spheres. This varied however with distance and quality of oil. It was also found that a previously impressed high voltage, raised the breakdown voltage for a sp. distance considerably. The easiest and most accurate measurements were obtained with 10 mm. balls. It was found that the com. transformer oils absorbed very little more H<sub>0</sub>O than did petroleum. Paraffin oil absorbs 0.003\%, petroleum 0.0005% and benzole 0.05% of H<sub>2</sub>O at 18°. A special arrangement, consisting of a cardboard-covered Fe tube filled with bitumen, or the like, and having a brass rod in the center, is recommended for testing breakdown voltage of substances used in cable covering. A method for detg. the energy losses in dielectrics is described. The first ammeter was made for measuring high frequency currents up to 40 amps. Cond. tests in Cu, Al and Fe show that there is a proportionality between the temp. coeff. and elec. cond. Expts. upon the influence of high voltage and the filling material on fuses show that there is less corrosion, due to overload, when the fuses are enclosed than when they are exposed, for the convection seems to cause oxidation. Various interesting results of fuse tests are given. The Pt and gas thermometers have been compared up to the m. p. of S and comparisons made between quartz and Jena glass containers. Work was begun upon the sp. heat of gases at low temps., using the method of Callendar and Barnes. An investigation of the narrowest bands of the spectrum of Fe, Ni, Mo, Pd, Si, Fe, U and W was made using the Wehnelt cathode. The construction of a bolometer for measuring absorption in the ultraviolet was started. The resistance increase in short wires due to rapid alternations was measured by alternately running a. c. and d. c. through a wire of 1 m. long which had a thermoelement fastened at the middle. When each gave exactly the same temp, the diff. in resistance was calc. from the inputs. Wire resistances of 1-100,000 ohms were found to have changed very little during the year whether kept in petroleum, paraffin oil, damp air, or hermetically sealed. Tests on Epstein samples of transformer Fe ran 3-4% lower than those made by the Nat. Phys. Soc. The Bismarkhütte tests show that the permeability may be greatest with samples cut 45° with the direction of rolling. The magnetic and elec. properties of 15 Fe-C alloys are being studied. Quartz-resistance thermos. were investigated and the Pt-resistance thermos. for low temps, checked within 0.1° at -78° and 0.2° at -190°. The different optical pyrometers tested check up to 1400°. Seger cones were found to soften in a ceramic oven as much as 100° below their rated temp. given by the Reichsanstalt. This is probably due to the increased length of time heated or to reducing gases present; at any rate it limits their use considerably. A method for the rapid testing of glass has been devized. The analysis of Au solns. is continued. Galalith as a substitute for rubber for insulation, was tested out and found to take up considerable moisture as well as to deteriorate with long use. W. E. RUDER.

Small Electric Furnaces with Heating Elements of Ductil Tungsten or Ductil Molybdenum. R. Winne and C. Dantsizen. J. Ind. Eng. Chem., 3, 770.—The general construction is an alundum (Al<sub>2</sub>O<sub>3</sub>), tube wound with either ductil Mo or W wire, which is packed in powdered SiO<sub>3</sub> in an air tight casing. H is led into the casing and diffuses through the alundum tube and burns at the mouth. Thus the Hipreserves the windings and at the same time protects the substance in the furnace from oxida-

tion. A crucible type is also discussed in which an alundum cylinder with a helical groove on its outer surface is used. The av. temp. of the furnace is 1650°.

G. F. VESSLER.

Production of Electric Steel. Anon. Elec. Rev. West. Elec., 59, 796.—The production of elec. steel has within the last years increased very rapidly throughout Europe and America. The total output in 1909 and in 1910, resp., for the U. S. was 22,947 and 55,355; for Germany, 17,773 and 36,188; for Austria-Hungary, 9,048 and 20,028 tons.

C. G. F.

The Direct Production of Molybdenum Steel in the Electric Furnace. E. F. Dittus AND R. G. Bowman. Colorado School of Mines. J. Ind. Eng. Chem., 3, 717-23; Trans. Am. Electrochem. Soc., 20, 135.—The authors describe their experimental furnace and the charges run in the same. The use of a sulfide ore of so active a metal as Mo in steel manuf. presents a number of problems. Foremost among these are the complete reduction of the Mo without serious loss, the diffusion of the Mo to form a homogeneous product and the elimination of S. The process employed was based on a reaction described by F. M. Becket (U. S. Pat., 855,157). For the reduction of MoS<sub>2</sub> in the presence of molten Fe some substance must be present of greater affinity for S than either Mo or Fe. Mn and Si seem best adapted. The authors conclude that; (1) Mo steel can be made in the elec. furnace by the direct reduction of Fe ore and the addition of MoS<sub>2</sub>, (2) Mo steel of low S content can be produced from MoS<sub>2</sub>, even in the form of low grade concentrate, by the use of Fe-Si as a desulfurizer. Recommendations as to the design of a furnace for small scale operations are given. Tar does not make a satisfactory binding material for crushed magnesite.

W. E. RUDER.

Application of the Electric Furnace to the Extraction of Zinc. F. C. Rass. mim., 35, 107.—The Snyder furnace, tried out in Switzerland and being set up in Nelson, Canada, requires but about 1 ton fossil C per ton of ore, is operated continuously, condenses the metal to the liquid state, forms an easily eliminated slag and affords great economy in labor.

Chas. A. Rouller.

Manufacture of Nitrogen Compounds by Electrical Power. E. KILBURN SCOTT. Electrician, 67, 886.—S. reviews the various elec. processes in industrial practice and the value of the products especially as fertilizers and as raw materials for the manuf. of explosives.

H. M. FERNBERGER.

The Electrolytic Production of Ozone Using Alternating Current. E. H. ARCHIBALD AND H. VON WARTENBERG. Z. Elektrochem., 17, 812.—Dil. solns. of H<sub>2</sub>SO<sub>4</sub> were electrolyzed with d. c. and then with a. c. superimposed. Owing to the depolarizing effect of the a. c. the output of O<sub>2</sub> was greatly increased over that obtained with d. c. alone—in some cases 300-fold. The electrodes were Pt. The max. output was obtained at about 6 amp., a. c., for all values of d. c. between 0.25 and 1.00 amp. An increase in the frequency was accompanied by an increase in O<sub>3</sub>. This depolarizing action of a. c. is applied by the Norddeutsche Affinerie in the Au-Ag parting process. Without the a. c., Cl is evolved at the anodes; with the superimposed a. c. all of the Cl combines with the Ag to form AgCl. Recently E. Buergin (Dissertation, Berlin, 1911) has succeeded in producing H<sub>2</sub>O<sub>2</sub> anodically with Zn electrodes; without the superimposed a. c., H<sub>2</sub>O and O are formed (see Ruer, Z. physik. Chem., 44, 81 (1903)).

Nickel Plating. P. S. Brown. Metal Ind., 9, 351.—A reply to criticism. Niplating solns. should be standardized. The double salts have no superiority over single salts for Ni-plating. If the double salts are decomposed, NH<sub>4</sub>OH is formed and there is danger of the soln. becoming alk. Since single salts are much more sol.

than the double they can be used at a greater d. and, since there is no NH<sub>4</sub>OH to decompose, at a higher efficiency.

W. E. RUDER.

The Pitting of Nickel Deposits and its Prevention. Anon. Brass World, 7, 316-7.—The pitting of the deposit is one of the difficulties encountered in Ni-plating. This is due to the formation of gas on the surface of the metal. The gas is due to:

(1) Too strong a current. (2) Lack of sufficient Ni in the soln. (3) Soln. too acid with H<sub>2</sub>SO<sub>4</sub>. A large quantity of H<sub>2</sub>BO<sub>2</sub> will overcome the pitting and produces a soft, white deposit. Agitation is also an excellent remedy. The addition of H<sub>2</sub>BO<sub>2</sub> to the plating soln. produces a white and soft deposit.

ROBERT KANN.

Mercurous Sulfate as Depolarizer in Standard Cells. G. A. HULETT. Princeton. Z. physik. Chem., 77, 411.—H. explains the misunderstanding of Van Ginnekins (C. A., 5, 1358) and differs from the conclusions there drawn. He points out that it is not certain that  $Hg_8SO_4$  follows the reaction  $Hg_8SO_4 + 2H_2O = Hg_8(OH)_2 + H_2SO_4$  and that these products are dissolved and dissociated in a CdSO<sub>4</sub> soln. W. E. RUDER.

Studies in Light Production. R. A. Houston. Electrician, 67, 800, 937, 966.—A series of papers dealing with the various com. sources of light. The theories involved are dwelt upon at length.

L. K. MULLER.

Corrections for the Effects of Atmospheric Conditions on Photometric Flame Standards. W. J. A. BUTTERFIELD, J. S. HALDANE AND A. P. TROTTER. Electrician, 67, 711; J. Gas Light., 115, 228; Chem. Eng., 14, 383-7: Engineering, 92, 199.

C. G. F.

How is Gas to be Compared with Electric Light from a Hygienic Point of View? K. SCHLESINGER. Elektrotech. Z., 32, 944—83.—S. seeks to disprove the statement made by gas engineers that gas lighting is more healthful than elec. lighting.

W. E. RUDER.

Metal Filament Lamps versus Central Stations in America. TSCHERNOFF. Elektrotech. Z., 32, 956.

W. E. R.

Mercury Arc with White Light. E. DARNOIS AND M. LE BLANC. Lumière elec., 15, 355-7.—A fluorescent reflector, made by depositing rhodamine on paper, is utilized successfully in the Cooper-Hewitt lamp to transform the disagreeable greenish light from an ordinary Hg lamp to light that is practically white. Rhodamine absorbs the yellow, green, violet, and ultraviolet rays and emits a red fluorescence (see also Elec. Rev. West. Elec., 59, 788).

C. N. MOORE.

Electrical Conductivity of Light Aluminium Alloys. ERNEST WILSON. Electrician, 67, 907.—A collection of certain light Al alloys are being weathered in order to investigate this influence upon the elec. cond. The Cu alloys deteriorate with the increase in % of Cu. The Cu-Mn alloy with 1.78% Mn has not seriously deteriorated in 10 yrs. exposure. The Cu-Ni alloy has been increased in resistance 18.7% in 10 yrs.

H. M. FERNBERGER.

Carbon Determinations in the Electric Furnace (AUGUSTIN). 7. Chloride Caustic-soda Plant (EBERT). 23. Dielectric Behavior of Porcelain (MINNEMAN). 19.

HOLTZ, A.: Ueber den Binfluss von Fremdstoffen auf Elektrolyteisen und seine magnetischen Bigenschaften. Berlin: E. Ebering. 8°, 2.40 M.

NERNST, W. AND BORCHERS, W.: Jahrbuch der Elektrochemie. Halle a/S.: 12.60 M.

- U. S., 1,001,105, Aug. 22. C. A. VON WELSBACH, Vienna, Austria-Hungary. Eliminating occluded gases from a filament containing osmium and at the same time forming an Os-alloy filament, by coating the filament with a magma containing metallic oxide (e. g., oxide of Th, Zr or Yt) in such proportion as to be reduced by the occluded gases, then expelling the gases by passing through the filament an elec. current, and finally raising the temp. of the filament to a point at which the oxide will be reduced by the gases and the metal caused to alloy with the Os. The Os may be partially replaced by Ru, Ir or Rh.
- U. S., 1,001,449, Aug. 22. J. H. ROBERTSON, New York, N. Y. Electrically extracting a metal from a solution containing powdered ore, by causing the continuous circulation of the soln. between 2 conducting surfaces to establish an elec. current to cause the metal to be deposited upon one of the surfaces and permit the tailings to remain in the soln.
- U. S., 1,001,497, Aug. 22. T. B. ALLEN, Niagara Falls, N. Y. Abrasive composition in homogeneous cryst. form, prepared by fusion in an elec. furnace and consisting principally of Al<sub>2</sub>O<sub>2</sub> and MgO, the MgO content not exceeding 25%.
- U. S., 1,001,570, Aug. 22. *Idem.* Abrasive composition, a homogeneous cryst. product formed by elec. m. with C a mixt. yielding oxides of Al, Mg and B in the proportions of 79%, 12% and 6%, resp., the remaining 3% being oxide of Fe, Si and Al.
- U. S., 1,001,571, Aug. 22. Idem. Abrasive composition, a homogeneous cryst. product consisting of Al<sub>2</sub>O<sub>3</sub> and BeO, the proportion of BeO being less than that represented by the formula BeOAl<sub>2</sub>O<sub>3</sub>, formed by elec. m. the oxides with C, reducing the impurities, allowing the fluid mass to cool and sepg. the cryst. product from the impurities.
- U. S., 1,001,572, Aug. 22. *Idem*. Treating crystallin fused alumina containing impurities (for use as an abrasive) with 2-5% NaCl at a high temp. (preferably 800-1200°) and removing the reaction products formed by the impurities and the NaCl.
- U. S., 1,001,589, Aug. 22. H. S. HATFIELD, Hove, England. Electrolytic transference cell, having an electrolyte in which the substance to be deposited in the cell is sol., 2 electrodes, one being porous and on one of which the substance is deposited and at the other the electrolyte becomes unsatd. with respect to the substance deposited, and a supply of the substance deposited resatg by contact therewith the liquid which has become unsatd.
- U. S., 1,001,770, Aug. 29. W. PFANHAUSER, Leipzig, Ger. Making electrolytic iron objects of good magnetic properties by depositing by electrolysis Fe products of larger dimensions than the finished size, reducing the products to the ultimate size by mechanical working and then annealing to restore the magnetic properties lost by the working.
- U. S., 1,001,876, Aug. 29. PAUL McDorman, Dayton, Ohio. Electrolytic cell for decomposing salt to form a bleaching liquid.
- U. S., 1,001,915, Aug. 29. L. YNGSTRÖM, Falu Grufva, Falun, Sweden. Electric furnace with a crucible-like working chamber and a stack-shaped charging chamber above the working chamber leaving a space between the walls of the 2 chambers at the mouth of the working chamber, and electrodes projecting into the working chamber in said space, the electrodes being held in position by the material to be treated, e. g., ore to be reduced, so that the material forms a substitute for a cover and the heatinsulating brick-work commonly used between the walls of the chambers.
- U. S., 1,002,249, Sept. 5. C. Ellis, Montelair, N. J. Oxidizing nitrogen by orming an elongated electric arc, passing a current of preheated air of const. moisture

- content concentrically about and in planes substantially at right angles to the normal axis of the arc and causing the arc to be diverted from its normal axis by magnetic action and to revolve about the normal axis in the path of the gaseous current at a speed less than that of the latter.
- U. S., 1,002,286, Sept. 5. A. R. LINBLAD, Ludvika, Sweden. Smelting iron, steel or other metals in a electric furnace, the elec. energy being supplied in the form of a 2-phase a. c. in such a way that the terminals of the one pole of both phases are each connected with 1 of 2 adjustable electrodes, hanging down into the furnace, from which electrodes the elec. current in the form of arcs passes to the smelting bath, the terminals of the other pole of the 2 phases being together connected with the conducting lining of the furnace or directly put in contact with the smelting bath.
- U S., 1,002,608, Sept. 5. F. J. Tone, Niagara Falls, N. Y. Assignor to Carborundum Co., same place. Treating emery ore, by forming a mixt. of pulverized ore with an amt. of C sufficient to reduce the constituents of the ore other than Al<sub>2</sub>O<sub>2</sub> and insufficient to reduce the Al<sub>2</sub>O<sub>3</sub>, interposing this mixt. as a resistance conductor between electrodes and passing an elec. current through it at a temp. below the reduction temp. of Al<sub>2</sub>O<sub>3</sub> and above the reduction temp. of SiO<sub>2</sub>, allowing the fused mass to cool and sepg. the reduction products from the fused Al<sub>2</sub>O<sub>3</sub>, an ingot of fused Al<sub>2</sub>O<sub>3</sub> being built up and fresh charge of mixt. supplied from time to time.
- U. S., 1,002,645, Sept. 5. W. T. CONN, Lakewood, Ohio. Assignor to National Carbon Co., Cleveland, Ohio. Negative electrode for flaming are lamps, having a main electrode portion (e. g., a C shell filled with C dust and water glass) and means for maintaining the arc at the end of the electrode comprizing a coating of insulating material (e. g., fire clay or asbestos) about the main portion of the electrode, and a shell of C surrounding the insulating material.
- U. S., 1,002,843, Sept. 12. A. HELFENSTEIN, Vienna. Electric furnace, having several sep. shafts, each shaft being arranged to work as an independent m. hearth, an electrode in each shaft and a single bottom electrode serving as common bottom electrode for all the shafts.
- U. S., 1,002,882, Sept. 12. W. B. THORPE, Balham, London, England. Electrolytic gas-liberating apparatus for use in meters, switches, etc., comprizing a main electrolytic cell and a high resistance arranged in series with each other and arranged to be connected in a shunt circuit across elec. supply mains, a main shunting resistance to be included in circuit with one of the supply mains and to one of which the electrolytic cell is connected, a second electrolytic or absorption cell connected in series with the high resistance and to the opposit end of the main shunting resistance, and a low resistance connected across the terminals of the absorption cell.
- U. S., 1,002,988, Sept. 12. A. HELFENSTEIN, Vienna. Utilizing gases evolved in electric furnaces, by introducing the charge into the elec. furnace so that it assumes a conical shape and at the same time surrounds and protects the upper electrode with freshly fed material, a space being provided about the conical mass into which gases generated during the reaction are discharged, air and combustible gases being admitted into the space to cause the combustion of the evolved gases so that the material freshly fed into the furnace is preliminarily heated in the upper zone of the conical charge. The patent also covers a furnace constructed to carry out the process.
- U. S., 1,002,989, Sept. 12. W. S. HELTZEN, Davis, W. Va. Electrolytic cell for decomposing salt, etc., with connections for passing an elec. current through the contents of the cell, a feed pipe entering the cell at its upper edge and extending to the bottom of the cell, the lower end of the pipe being at an angle, an outlet pipe at the opposit end of the cell and entering the end wall of the cell near its upper edge, horizon-

- tal plates extending inwardly from the side walls and carrying vertical flanges and provided with openings intermediate their edges, a comp. filling on the inner faces of the side walls, enveloping about 1/3 of the plates, a metallic strip resting squarely against the inner edges of the plates and being braced by them, a removable diaphragm secured against the metallic strip and connections for passing an elec. current through the diaphragm and strip.
- U. S., 1,003,041, Sept. 12. E. G. EKSTROM, Los Angeles, Cal. Making alkali hydroxides and their derivatives by electrolysis of a soln. of an alkali-F compd., such as Na fluosilicate between a C anode and a metallic cathode, e. g., Hg. Graphite electrodes sepd. by a diaphragm may also be used.
- U. S., 1,003,092, Sept. 12. H. H. Dow, Midland, Mich., and W. S. GATES and A. B. SCHABFER, Worthington, Ontario, Canada. Assignors to Ontario Nickel Co., Ltd., Worthington, Canada. In a continuous process of recovering nickel from nickel sulfate, electrolyzing a soln. of the sulfate, agitating the resulting acid electrolyte with an excess of basic Ni ppt. apart from the electrolytic cell to refine and reduce the Ni, settling the mixt. thus formed, returning the supernatant neutral electrolyte to the cell to be further electrolyzed, and returning the settlings of basic Ni ppt. to neutralize the acid electrolyte again produced.
- U. S., 1,003,187, Sept. 12. A. LEUCHTER, Brooklyn, N. Y. Making an electrotype by electrolytically depositing upon the conducting surface of a mold an alloy of Ni and Fe and then applying a backing of another metal (e. g., Cu) to the deposited alloy; the Ni in the process being employed as anode and the Fe salt in soln.
- U. S., 1,003,299, Sept. 12. O. SCHÖNHERR, Christiania, and J. HESSBERGER, Christianssand, Norway. Maintaining a long stable electric arc (e. g., in causing reactions between gases) by means of a current of gas, that end of the arc at or near which the gas is admitted into proximity to the arc being caused to move upon the surface of a hollow electrode while the greater portion of the arc remains stationary, and a portion of the gas being caused to pass through the hollow electrode.
- U. S., 1,003,354, Sept. 12. W. T. Gibbs, Buckingham and R. A. Witherspoon Shawinigan Falls, Quebec, Canada Assignors to Shawinigan Carbide Co., Ltd., Montreal, Canada. Electrode for electric furnaces, having a head of conducting metal with several screw-threaded sockets, graphite plugs or short rods screwed into the sockets and presenting interiorly screw-threaded recessed ends, amorphous C rods screwed at one end into the recessed ends of the plugs or short rods, a substance conductive of electricity, e. g., a baked product of coke, tar and pitch, incasing the pencils thus formed and a sheet Fe shell enclosing the conductive casing and rods.
- U. S., 1,003,374, Sept. 12. W. SCHLOEMILCH, Berlin, and P. F. PICHON, Steglitz, Ger. Wave detector for wireless telegraphy, consisting of 2 contact bodies, one of Cu pyrites and the other of Pt.
- U. S., 1,003,375, Sept. 12. Idem. Wave detector for wireless telegraphy, consisting of 2 contact bodies, one of Fe pyrites and the other of Pt.
- U. S., 1,003,456, Sept. 19. L. H. A. B. M. HAZARD-FLAMAND, Boulogne-sur-Seine, France. Assignor to International Oxygen Co. Electrolytic apparatus for producing oxygen and hydrogen, having a diaphragm between the electrodes, a fluid seal about both sides of the top of the diaphragm, the inner of the seals being double, connections for constantly delivering the material to be decomposed, e. g., H<sub>2</sub>O, to one portion of the double seal, one of the seals being in communication with the other and the other having an opening communicating with opposit sides of the diaphragm so that the material is caused to circulate from one portion of the double seal to the other before being discharged about the electrodes.

- U. S., 1,003,659, Sept. 19. F. J. Root, New York, N. Y. In making electroplated wire cloth, passing in a continuous path a long strip of woven wire cloth alternately over and under upper and lower rollers and electrically depositing the plating material on the cloth while in the bath in which the lower rollers are located, the upper rollers being above the bath and anodes being placed between the folds of the material.
- U. S., 1,003,789, Sept. 19. V. Popp and A. Miner, Paris, France. High-temperature resistance furnace having an outer wall forming the furnace chamber, a tank therein, a resister of granular material in the chamber, a protecting wall or plate between the tank and resister, and a protecting wall between the resister and the wall of the furnace chamber, the protecting walls being made of carborundum.
- U. S., 1,003,799, Sept. 19. A. RODECK, Milan, Italy. Electrolytically forming hollow articles by successively depositing given quantities of metal on one portion of a moving cathode; forming the article by simultaneously and successively plating out the same quantities of metal from another portion of the cathode; moving the article during the plating operation, and simultaneously depositing metal on its interior.

Brit., 17,469, July 22, 1910. FRITZ EISNER, 341-5 Birkbeck Bank Bldgs., London. In the manuf. of metallic filaments from W or other refractory metals or compds. thereof, a binding medium devoid of C and composed of hydrazine or its compds.

Brit., 20,337, Aug. 31, 1910. P. F. COWING, 10 W. 107th St., New York. Eliminating copper from alloys contg. Ni and Cu, by bringing the original alloy into soln. electrolytically, substituting Fe for Cu in this soln., and electrolytically depositing Ni and Fe from the soln. thus obtained.

# 5. PHOTOGRAPHY.

LOUIS DERR.

New Developers. J. Desalme. Bull. soc. franc. phot., [3] 2, 291.—p-Amino-p-hydroxydiphenylamine, diaminohydroxydiphenylamine hydrochloride, and triaminodiphenylamine hydrochloride possess the property of regenerating themselves by addition of Na<sub>2</sub>SO<sub>2</sub>. An excellent developing bath is: p-phenylenediamine 10 g., NaHSO<sub>2</sub> (35° Bé.) 25 cc., NaOH (40° Bé.) 40 cc., KBr 3 g. This keeps indefinitly out of contact with the air and absorbs atmospheric O only slowly. [Note by L. D.: no water is given in the recipe, though the bath obviously requires considerable dilution.]

The Krayn Process. H. Quentin. Bull. soc. franc. phol., [3] 2, 293-300.—The new Krayn screen is prepared as follows: on a gelatin surface a line or other geometric pattern is printed in fatty ink, and the plate immersed in a dye, e. g., scarlet-red G, which colors the unprinted areas. A bath in FeCl, mordants the dye and tans the gelatin enough to make it repellant to further dye applications. The ink is removed with turpentine and a second design printed crosswise on the plate in fatty ink. Immersion in methylene blue MLB (Hoechst) dyes the uninked portions of the plate, but not the inked areas nor the repellant red-dyed spots. The ink is again removed and the dye mordanted as before, and the plate is immersed in a mixture of Hoechst blue A and yellow F, which fills the remaining areas with green color. The plate is then coated with a pinachrome panchromatic emulsion and manipulated like other similar plates. The screen contains 250 elements per sq. mm., as compared with 7000 for the autochrome and 775 for the omnicolor plates. Full working directions are given.

Theory and Practice of Intensifying. A. SCHULLER. Phot. Rundschau, 25, 141-5, 178-81; cf. C. A., 5, 3202. — By measuring the gradation-curves of treated and untreated plates it was shown that in general intensifiers give equal intensification to all tones, except in cases where the intensification is a purely photographic process rather than a chemical one. At the beginning, or when the intensifier diffuses slowly, there is a slight surface deposit which darkens the shadow-regions proportionally more than the lights. To obtain equal intensification the HgBr, or U intensifier should be used; to intensify the shadows more than the lights, use the Hg intensifier or the U intensifier with short time of application. The author also gives a Au intensifier, as follows: 1% AuCl<sub>2</sub> 15 cc., 10% CuSO<sub>4</sub> 6 cc., 10% NaCl 2 cc.; this gives a strong surface intensification only.

Comparative Studies on Photographic Reducers and Intensifiers. E. STENGER. Z. Reprodukt., 13, 70-5; cf. C. A., 5, 3382.—Bromides and iodides behave in general like chlorides, but fluorides do not show the characteristics of the other halogens. Plates reduced with persulfate lose density in the fixing bath without change of gradation, and similarly gain density in a developing bath.

L. DERR.

Automatically Obtaining the Characteristic Curve. E. Goldberg, R. Luther And F. Weigert. Z. wiss. Phot., 9, 323-31.—A neutral gray photometric wedge is laid against the plate to be tested, an exposure given and the plate developed and fixed as usual. After drying, plate and wedge are laid together crosswise, whereupon the form of the characteristic exposure-density curve can be seen by transmitted light. It agrees well with the results of the more elaborate photometer measurements. In the absence of a wedge, a substitute can be made from superposed strips of paper, though paper showing no selective absorption is quite difficult to obtain. L. Derg.

Photochemical Studies. II. Classification of Light Reactions. I. PLOTNIKOV. Z. physik. Chem., 77, 472-81.—A systematic arrangement, in tabular form, of the known irreversible light-reactions. It is based on the two general principles of light-reaction: that it may progress only under the light, ceasing in darkness, or that there may be a reaction in the dark quite different from this, in which case the result of a chem. process is the sum of the 2 reactions. Three classes of reaction are distinguished: purely photocatalytic action, without action in the dark or continuating action; purely photochem. action, taking place only in presence of a catalyzer and in the light; indirect photochem. catalytic action, the action beginning in presence of a catalyzer and in the light and continuing at the same rate in darkness. Subdivisions and combinations of these 3 classes are made, and examples of each are given when known to exist.

L. Derr.

Reduction of Exposure with Autochromes. A. VON PALOCSAY. Wiener Mitt., June 10, 1911, 19-21; June 25, 10-15.—P. increased the sensitiveness 3-fold by immersing the plates 2 min. in darkness in the following bath: 200 cc. distilled water, 50 cc. abs. alc., 2 cc. alkaline 1: 500 pinachrome (Hoechst). Drying was in darkness as quickly as possible. The light-filter was composed of 100 cc. distilled water, soft gelatin 10 g., aesculin 0.5 g., poured on glass, 1 cc. per cm. of filter surface. L. D.

Pictures and Decorations on Glass. 19.

EDER, J. M.: Jahrbuch für Photographie und Reproduktionstechnik, 1911. Halle a/S. 9.50 M.

WOLF-CZAPEK, K. W.: Angewandte Photographie in Wissenschaft und Technik. (4 Teile.) Teil I. Die Photographie im Dienste der anorganischen Naturwissenschaften. Berlin 8°, 100 pp., 4.50 M.

U. S., 1,002,386, Sept. 5. Z. J. Gold, Stella, and W. M. Newton, Granby, Mo. Making black and white negatives for photo-engraving, etc., by applying to an extra fast gelatin portrait dry plate after exposure in a camera, a developer composed of dist. H<sub>2</sub>O 6 oz., hydroquinone 24 g., Na<sub>2</sub>SO<sub>2</sub> crystals 180 g., K<sub>2</sub>CO<sub>2</sub> 120 g., sugar ½ teaspoonful, Na hyposulfite 48 g., and then applying a fixing bath composed of dist. H<sub>2</sub>O 128 oz., H<sub>2</sub>SO<sub>4</sub> 2 drams, Na<sub>2</sub>SO<sub>3</sub> 1.5 oz., chrome alum 1.25 oz. and Na hyposulfite 2 lbs.

U. S., 1,003,720, Sept. 19. L. DUFAY, Chantilly, France. Assignor to Soc. An. des Plaques & Produits Dufay, same place. Making homogeneous polychrome screens for color photography by covering in lines of greasy material the surface of a layer of transparent material, e. g., gelatin-coated glass or film, adapted to be colored by imbibition, so as to leave free the surface to receive the first tint, dyeing this portion, covering the whole with a varnish having a resinous base which only adheres to the colored part so as to protect it from subsequent coloration, the varnish being of such comp. that the solvent therein does not dissolve the greasy material while the resin is insol. in a solvent of the greasy material, e. g., benzine, and not adapted to mix with the greasy material, removing the varnish by friction where it covers the greasy material, removing the latter by a solvent, e. g., benzine or turpentine, making a second similar application of greasy material in lines crossing the lines of the first application, coloring the free surface by imbibition with the second color, varnishing, removing the varnish over the greasy lines, dissolving out the latter, coloring the remaining surface by imbibition with the third color and repeating the operation for each additional color desired.

### 6. INORGANIC CHEMISTRY.

#### H. I. SCHLESINGER.

Sulfur-Phosphorus Compounds. J. Mai. Univ. Bern. Ber., 44, 1229–33; cf. C. A., 5, 1395, 1936.— $P_7S_a$  and  $P_4S_b$  are prepared by heating  $P_4S_a$  with S at 175–80°, in the presence of naphthalene. At this temp.,  $P_4S_b$  is sepd., while  $P_4S_{10}$  crystallizes ou ton cooling. The heating is best carried out in a current of CO<sub>2</sub>. Excess of S and  $P_4S_a$  are removed by liquid naphthalene.

Sulfur-Phosphorus Compounds. II. Syntheses with Yellow Phosphorus. J. Mai. Univ. of Bern. Ber., 44, 1725-7; cf. preceding abstr.—A soln. of the elements in  $CS_2$  was added to naphthalene at 110° and the temp. slowly raised to 195°. Upon cooling, crystals of  $P_4S_7$  or  $P_4S_{10}$  sepd., according to the proportions taken. A soln. of P and S in xylene boiled for 30 min., with occasional addn. of a trace of I, gave  $P_4S_7$  only, even when S was in large excess. Attempts to prepare  $P_4S_2$  in xylene and in naphthalene gave only  $P_4S_7$ .  $P_4S_2$  has been prepared by the author.

A. R. MIDDLETON.

The System: Chromic Oxide, Water. R. KREMANN. Univ. Graz. Monatsk., 32, 619-22.—A table and curves are given for the system  $CrO_2$ - $H_2O$ . When the conc. of the  $CrO_3$  ranges from 0 to 57.2%, ice alone is sepd.; when the % of the  $CrO_3$  increases from 57.2% to 71.2% anhydrous  $CrO_3$  is sepd. No hadrate of  $CrO_3$  is formed.

Contributions to the Knowledge of Holmium. O. HOLMBERG. Archiv. Kemi. Min. Geol., 4, 2, 8.—Investigations were based on 29 kg. of euxenite. The sample was treated with conc. H<sub>2</sub>SO<sub>4</sub> and the ppt., after adding NH<sub>4</sub>OH to the resulting H<sub>2</sub>SO<sub>4</sub> soln., digested with hot conc. HNO<sub>2</sub>. The oxide resulting from ignition of the ppt. obtained through treating the HNO<sub>3</sub> soln. with H<sub>2</sub>C<sub>2</sub>O<sub>4</sub> weighed 5.5 kg. Hot conc.

(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> took up about 700 g. of this, and fractional crystn. was then instituted. Holmium was found to lie midway in the range of solubilities of those of Yt and Eb. A salt perfectly free from Yt, Tb, and Dy could not be obtained. Accordingly, whereas the at. wt., as computed directly, would be 150, H. fixes it at 156.

C. G. GILBERT.

Alleged Complexity of Tellurium. A. G. V. HARCOURT AND H. B. BAKER. Christ Church, Oxford. J. Chem. Soc., 99, 1311-13; Proc. Chem. Soc., 27, 187.—The method of fractional pptn. of TeCl<sub>4</sub>, applied by Browning and Flint (C. A., 3, 2913; 4, 3049), was used, but the mean value found for the at. wt. of Te was 127.54 as compared with 127.53, found by omitting the fractional pptn. The authors ascribe the low at. wt. found by B. and F. to the presence of impurities.

D. M. LICHTY.

Some New Inorganic Salts. T. V. BARKER. Mineralog. Dept., Univ. Museum, Oxford. J. Chem. Soc., 99, 1326-9; Proc. Chem. Soc., 27, 198.—The method of prepn. and analysis, the cryst. form (monoclinic) and crystal measurements of 2 new salts,  $Rb_2Mg(CrO_4)_2$ . $6H_2O$  and  $Cs_2Mg(CrO_4)_2$ . $6H_2O$ , are given. They are isomorphous with each other, with the corresponding NH<sub>4</sub> salt (Murmann, Sitzb. K. Akad. Wiss., Wien., 27, 175 (1857)) and with the double sulfates of similar comp.  $LiIO_4.H_4O$  was obtained in the form of simple hexagonal prisms, and is not isomorphous with the periodates of Na, K, Rb, Cs and NH<sub>4</sub> (cf. J. Chem. Soc., 93, 15; Rammelsberg, Pogg. Ann., 134, 389, 1868).

D. M. LICHTY.

Theory of Platinum Catalysis in the Oxidation of Hydrogen with Sulfuric Acid.

J. Milbauer. Prague, K. K. böhm. techn. Hochschule. Z. physik. Chem., 77, 380-4.

—First is given a summary of the hypotheses put forward to explain the catalytic action of Pt. Since the max. action of Pt in the oxidation of H, at 174°, occurs with H<sub>2</sub>SO<sub>4</sub> satd. with a sol. Pt. salt, probably a sulfate, the catalysis is here ascribed to the intermediate formation of a Pt compd. Solns. of Pt black gave the best results. The catalysis is slowly poisoned by As<sub>2</sub>O<sub>3</sub>.

D. M. LICHTY.

Cobaltic Nitrites. ARTHUR ROSENHEIM AND A. GARFUNKEL. Berlin. 44, 1865-73.—In investigating the relations of the guanidinium triaquocobaltic trinitrites described by K. A. Hofmann and Buchner (C. A., 4, 172) the authors prepared the compds. by adding guanidine carbonate to a conc. soln. of Na<sub>2</sub>Co(NO<sub>2</sub>)<sub>6</sub>. One mol. of carbonate to 1 of nitrite formed (CN<sub>2</sub>H<sub>6</sub>)<sub>2</sub>Na[Co(NO<sub>2</sub>)<sub>6</sub>]; 3 mols. of carbonate to 1 of nitrite formed (CN<sub>2</sub>H<sub>2</sub>)<sub>2</sub>[Co(OH)<sub>2</sub>(NO<sub>2</sub>)<sub>2</sub>], granit-red; agreeing in all properties with the second salt of H. and B., but containing no Na. From the mother liquor small brick-red needles were obtained, agreeing in properties and comp. with (CN<sub>2</sub>H<sub>6</sub>)<sub>2</sub>-[Co(OH)<sub>2</sub>(NO<sub>2</sub>)<sub>2</sub>] described by H. and B. These two salts differ in appearance, solubility and some pptn. reactions and are probably stereoisomers. The first gives with TINO<sub>2</sub> cinnabar-red crystals of difficultly sol. Tl<sub>2</sub>H[Co(OH)<sub>2</sub>(NO<sub>2</sub>)<sub>3</sub>], stable even in b. H<sub>2</sub>O; the second gives Tl<sub>2</sub>(CN<sub>2</sub>H<sub>6</sub>[Co(OH)<sub>2</sub>(NO<sub>2</sub>)<sub>3</sub>], brown-red, easily sol., decomposed by H<sub>2</sub>O with pptn. of Co<sub>2</sub>O<sub>2</sub>. If the aq. soln. is treated with AcOH and warmed, the cinnabar-red, guanidine-free salt is pptd. A previously prepared Zn salt is now proved to belong to this series and to have the formula ZnH[Co(OH),(NO,),].4H,. It gives with TlNO<sub>2</sub> and with AgNO<sub>2</sub> the corresponding salts. SrH[Co(OH)<sub>2</sub>(NO<sub>2</sub>)<sub>4</sub>].-4H<sub>2</sub>O, granit-red, sol. in H<sub>2</sub>O, unstable in soln. or dry, was prepared. By the action of Na acetylacetonate on Na<sub>2</sub>Co(NO<sub>2</sub>)<sub>0</sub>, Na[Co(C<sub>5</sub>H<sub>7</sub>O<sub>2</sub>)<sub>2</sub>(NO<sub>2</sub>)<sub>2</sub>].<sub>5</sub>H<sub>2</sub>O, purple-red, was obtained. The H<sub>2</sub>O can be withdrawn over H<sub>2</sub>SO<sub>4</sub> without decomposing the salt. At 30° it crystallizes without H<sub>2</sub>O in bright-red needles. Its aq. soln. with salts of all other alkali metals forms  $Me[Co(C_0H_7O_2)_2(NO_2)_2].H_2O$ ; the Tl salt is anhydrous; dil. aq. soln. with dil. AgNO<sub>2</sub> gives Ag[Co(C<sub>1</sub>H<sub>2</sub>O<sub>2</sub>)<sub>2</sub>(NO<sub>2</sub>)<sub>3</sub>], red-brown; conc. soln. with conc. AgNO<sub>2</sub> forms  $Ag_1[Co(C_2H_7O_2)_2(NO_2)_2]$ , dark yellow. On b. the Na salt soln., brown flocculent decomp. products are formed, then dark-green crystals of

 $Co(C_aH_rO_a)_a$ . By adding to the mother liquor from the Na salt, 2 mols. Na acetylacetonate, a salt of divalent Co results,  $Na[Co(C_aH_rO_a)_a]$ . The authors prepared also Zn and Cd acetylacetonates not previously known.

A. R. MIDDLETON.

The Asymmetric Cobalt Atom. A. WERNER. Univ. of Zurich. Ber., 44, 1887-98.—According to the coordination theory, compds. of [Ame en] contain an asymmetric central atom and should exhibit optical isomerism when A and B are in the cis position. With the aid of d-bromocamphorsulfonates the author has succeeded in sepg. the active forms of 1-chloro-2-aminodiethylenediamino-cobaltic bromide, [ClNH,Co(en), ]Br., and of 1-bromo-2-aminodiethylenediamino cobaltic bromide, [BrNH<sub>2</sub>Co(en)<sub>2</sub>]Br<sub>2</sub>. The chloride of the former was treated with Ag d-bromocamphorsulfonate, the d-form is less sol. and crystallizes first;  $[\alpha]_c = +69.5^\circ$ ; for the *l*-form,  $[\alpha]_c = +31.25^{\circ}$ . Both the *d*- and *l*-sulfonates were transformed to dithionates by Na,S,O, and these by rubbing up with conc. HBr, to the bromides. The d-sulfonate can be changed directly to bromide by rubbing with conc. HBr;  $[\alpha]_c =$ +43.1°; for the l-bromide  $[a]_c = -43.0^\circ$ . dl-1-Bromo-2-aminodiethylenediamine cobaltic bromide, [1Br2NH2Co(en),]Br, (prepared by 2 different methods) was treated with aq. soln. of aq. d-bromocamphorsulfonate; the d-form crystallized first;  $[\alpha]_c =$ +65.7°; from the mother liquor the dl-dithionate was pptd. by Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>, and, by adding more Na,S,O, the l-dithionate; the d-sulfonate and the l-dithionate with conc. HBr gave the corresponding bromides;  $[\alpha]_c = +45.0^\circ$  and  $-45.0^\circ$ . Contrary to expectation, the active forms proved remarkably stable. Solns. of the bromides of the bromo series did not racemize after long standing or even brief b. and these salts were successively transformed into various salts without loss of activity.

A New Method for the Separation of Cerium. C. James and L. A. Pratt. New Hampshire Coll. J. Am. Chem. Soc., 33, 1326-30.—Ce is pptd. completely as basic ceric nitrate and bromate, free from other earths, by b. the rare earth nitrates with KBrO<sub>2</sub> in presence of a lump of marble.

A. R. MIDDLETON.

New Rare Barth Compounds. L. A. PRATT AND C. JAMES. New Hampshire Coll. J. Am. Chem. Soc., 33, 1330-2.—Several new compds. of Y, Sm, Nd, Pr, La, Ce, and Th with organic acids are described. Th can be sepd. from the rare earths by phenoxyacetic acid.

A. R. MIDDLETON.

Thulium. I. C. James. New Hampshire Coll. J. Am. Chem. Soc., 33, 1332–44.—The extraction of Tm from various minerals is described. Tm cannot be sepd. into simpler substances. Several salts are described.

A. R. MIDDLETON.

The Oxidation of Hydrazine. V. Reaction between Potassium Iodate and Hydrazine Sulfate. C. F. Hale and H. W. Redfield. Cornell Univ. J. Am. Chem. Soc., 33, 1353-62.—The reaction proceeds quantitatively according to the Rimini equation:  $5N_2H_4.H_2SO_4 + 4KIO_2 = 5N_2 + 12H_2O + 2K_2SO_4 + 3H_2SO_4 + 2I_2$ . The reaction is the same whether  $N_2H_4$  is added to  $KIO_2$  cold (atm. of  $CO_2$ ) or at b. temp. (atm. of steam).

A. R. MIDDLETON.

Europium. C. James and J. E. Robinson. New Hampshire Coll. J. Am. Chem. Soc., 33, 1363-5.—The extraction of Eu from very large amts. of material is described.

A. R. MIDDLETON.

Melting Temperature of Sodium and Lithium Metasilicates. F. M. JAEGER. J. Wash. Acad. Sci., 1, 49–52.—Pure Li<sub>2</sub>SiO<sub>2</sub> was prepd. by fusing together Li<sub>2</sub>CO<sub>3</sub> and quartz, grinding, re-fusing, adding more Li<sub>2</sub>CO<sub>2</sub> until a homogeneous product having the desired proportions was obtained. Li<sub>2</sub>SiO<sub>3</sub> crystallizes in white glistening needles, n in direction of elongation, 1.609, perpendicular, 1.589, birefringence 0.019,  $d_{M'9} = 2.5203$ , m. 1201.8° (sharply). The solidifying temp. is variable, dependent on the rate of cooling. The same is true of Na<sub>2</sub>SiO<sub>3</sub>, which crystallizes much more slowly

This probably explains the discordant results found in the literature for its f. p. The m. p. of Na<sub>2</sub>SiO<sub>3</sub> is 1088°, n about 1.527-1.518, birefringence about 0.01.

GEORGE W. MOREY.

Silver Fluoride and Silver Subfluoride. L. Vanino and Paul Sachs. Kgl. Akad. Wiss. Munich. Z. anal. Chem., 50, 623-9.—When a conc. soln. of AgF (prepd. from Ag<sub>2</sub>CO<sub>2</sub> and HF) is evapd. in vacuum, dark brown crystals are formed on the surface, while large clear tetragonal ones are formed in the liquid. When a dil soln. is allowed to stand in a desiccator at atm. pressure, the brown crust is not formed, but a mass of parallel colorless crystals ppt. When these crystals are kept in a H<sub>2</sub>SO<sub>4</sub> desiccator they turn yellow on the surface. Analysis of the products in both cases gave the comp. AgF.H<sub>2</sub>O. The brown color in the above and in com. preps. is due to colloidal Ag<sub>2</sub>O. AgF is insol. in organic solvents, and its aq. soln. is neutral. Attempts to prepare the Ag<sub>2</sub>F of Guntz only resulted in a product containing AgF, Ag<sub>2</sub>O and Ag.

GEORGE W. MOREY.

Lithium Amalgams. G. Y. Zhukovskii. Polytechn. Inst. Kaiser Peters des Grossen, St. Petersburg. Z. anorg. Chem., 71, 403-18.—The system Li-Hg was studied by taking cooling curves of mixts. fused in Fe tubes, and protected from oxidation by a covering of fused salts or paraffin. The m. p. of Li (179°) is lowered by addition of Hg to the eutectic at 2.4 at. % Hg and 162°. The fusion curve then rizes, corresponding to the compd. LinHg. Melts between 2.4 and 24.8 at. % Hg show a transformation at 166°, indicating a polymorphous modification. This branch of the curve shows a break at 16.4 at. % Hg and 275°, due to formation of a compd. of unknown comp. which decomposes at its m. p. The fusion curve rizes regularly to 25 at. % Hg and 379°, at which temp. all melts between 25 and 33 at. % Hg begin to crystallize, though the point is found with melts up to 39.9 at. % Hg. This may be due to a compd. Li<sub>n</sub>-Hg<sub>2</sub> (n = 3 or 4) possessing a very low heat of fusion, but its existence is doubtful. The fusion curve then rizes to 600.5°, the m. p. of the compd. LiHg, after which if falls to 338°, and 69.9 at. % Hg, where it shows a break, due to the compd. LiHg, which melts with decomp. LiHg forms cubic crystals, hardness 2-3, and forms solid solns. with both Li and Hg, between the limits 39.9-61.8 at. % Hg. A break in the curve at 82-4 at. % Hg and 232°, indicates the compd. LiHg, which melts with decomp. The fusion curve falls rapidly to the eutectic between 99.03 and 100% Hg and -42°, then rizes to the m. p. of Hg. The existence of the compds. LiHg and LiHg, was confirmed by calorimetric measurements (method of Berthelot). The ht. of formation of LiOH aq. was found to be 52.7 cal., of Li<sub>2</sub>SO<sub>4</sub> aq., 133.7 cal. Z. compares the compds. formed by Li, Mg, Na, K, and Cs with Zn, Cd and Hg, and concludes that Li resembles Mg more than the alkali metals. (Also in J. Russ. Phys. Chem. Soc., 43, 807-25.) GEORGE W. MOREY.

Persulfates of Divalent Metals. G. A. BARBIERI AND F. CALZOLARI. Chem. Inst., Univ. Ferrara. Z. anorg. Chem., 71, 347-55.—Cryst. persulfates of the divalent metals are difficult to obtain because of their great solubility and instability. By replacing H<sub>2</sub>O of crystn. by NH<sub>2</sub>, Py, and C<sub>0</sub>N<sub>4</sub>H<sub>2</sub>, the author obtains stable compds. By double decomp. between a conc. soln. of (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub> and a conc. NH<sub>2</sub> soln. of the metal sulfate the corresponding NH<sub>2</sub> compd. seps. as a cryst. ppt. Tetrammine-zinc persulfate, ZnS<sub>2</sub>O<sub>8</sub>.4NH<sub>2</sub>, large transparent prisms, ppts. only from very conc. solns. Hexammine-cadmium persulfate, CdS<sub>2</sub>O<sub>8</sub>.6NH<sub>3</sub>, forms white microscopic crystals, less sol. than preceding. Strong NH<sub>3</sub> odor, decomp. on standing in moist atm. Hexammine-nickel persulfate, NiS<sub>2</sub>O<sub>8</sub>.6NH<sub>3</sub>, clear blue-violet crystals, strong NH<sub>3</sub> odor. Becomes black in moist air; H<sub>2</sub>O soln. is blue, turns cloudy, ppts. NiO<sub>2</sub>. When dried over CaCl<sub>2</sub> it turns gray, then green, with loss of NH<sub>2</sub> and O, and forms a double sulfate. In vacuum over H<sub>2</sub>SO<sub>4</sub> it turns dirty yellow. Tetrammine-copper persulfate, CuS<sub>2</sub>O<sub>8</sub>.

aNHa, dark blue-violet needles, more stable than the preceding. All the above compds. lose NH<sub>2</sub> and O on standing some time, and explode when strongly heated or when struck. They are sol. in H<sub>2</sub>O, from which soln. BaCl<sub>2</sub> only produces a slight cloudiness. The H<sub>2</sub>O soln. decomposes slowly with deposition of a basic salt containing much sulfate. The Pv compds, are similarly prepd., using 8-10 mol. Pv to 1 of metal sulfate. Tetrapyridine-sinc persulfate, ZnS<sub>2</sub>O<sub>4</sub>.4Py, glittering prisms, forms slowly from very conc. soln. Decomp. readily, turning yellow. Tetrapyridine-cadmium persulfate, CdS<sub>2</sub>O<sub>2</sub>.4Py, sharp glittering needles, less sol. than the Zn salt. Tetrapyridine-nickel persulfate, NiS<sub>2</sub>O<sub>4</sub>.4Py, thin, clear, blue microscopic needles, forms only from very conc. solns. Tetrapyridine-copper sulfate, CuS<sub>2</sub>O<sub>8</sub>-4-Py, masses of blueviolet microscopic needles, insol. in presence of excess Py and (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>2</sub>. Hexamethylenetetrammine compds. were similarly prepd., using 2 mol. C<sub>2</sub>H<sub>12</sub>N<sub>4</sub> to 1 mol. metal sulfate. The compds, obtained should be quickly sepd, and washed with EtOH, since they decomp. rapidly when moist but are more stable when dry. Bishexamethylenetetrammine-magnesium persulfate-octahydrate, MgS<sub>2</sub>O<sub>2</sub>.8H<sub>2</sub>O.2C<sub>2</sub>N<sub>2</sub>H<sub>12</sub>, forms colorless silky scales. Bishexamethylenetetrammine-manganese persulfate-octahydrate, Mn.S.O. 8H<sub>2</sub>O.2C<sub>a</sub>N<sub>4</sub>H<sub>132</sub> colorless scales. Bishexamethylenetetrammine-cobalt persulfate-octahydrate, MnS<sub>2</sub>O<sub>4</sub>.8H<sub>2</sub>O.2C<sub>4</sub>N<sub>4</sub>H<sub>19</sub>, rose-colored scales. Bishexamethylenetetrammine-nichel persulfate-octahydrate, NiS<sub>2</sub>O<sub>2</sub>.8H<sub>2</sub>O.2C<sub>2</sub>N<sub>2</sub>H<sub>10</sub>, green scales. The above 4 compds. are all isomorphous. GEORGE W. MOREY.

Uranium Hexafluoride. Otto Ruff and Alfred Heinzelmann. Danzig. Z. anorg. Chem., 72, 63-84.—When UCla is treated with F, UFa UFa and Cla result. The reaction is carried out in a special app., and after decomp, is complete the UF. is sepd. from UF, by sublimation. From the fact that both UF, and UF, are formed, the authors suggest that UCl, has the constitution UCl, UCl,. When UCl, is boiled with anhydrous HF until HCl no longer is given off, and the HF removed by distillation, the residue obtained is probably UF.HF. When this is heated HF is first evolved, then UF, leaving a residue of UF. UF, is also obtained by treating U or UC, with F, in the presence of Cl, which acts as catalyzer. The yield with U is 85-94% of the theoretical. UF, forms pale yellow glittering monoclinic crystals which are strongly hygroscopic. The best solvent for it is C<sub>2</sub>Cl<sub>2</sub>; others are CCl<sub>2</sub>, CHCl<sub>2</sub> and C<sub>2</sub>H<sub>2</sub>NO<sub>2</sub>, but it reacts with other organic solvents. It reacts energetically with H<sub>2</sub>O, H and other reducing agents of all kinds, generally giving UF4. With S a new compd. is formed, a colorless gas of unknown comp., m. p. -135°, b. p. -40°. The b. p. of UF<sub>4</sub> is 56° at 760 mm., the m. p. 69.5°, the sp. gr. 4.68 at 20.7°, the mean heat of vaporization between 37-56.2° (calc. from v. p. detns.) 29.4 cal. per g.

GEORGE W. MOREY.

The Rate of Hydration of Metaphosphoric Acid. D. BALAREFF. Chem. Lab. Staatsgym. Rustschuk. Z. anorg. Chem., 72, 85-8; cf. C. A., 5, 41.—Montemartini and Egidi (Chem. Zentr., 1901, II, 391) found that the rate of hydration of HPO3 increased with increasing acid conc., and suggested that the hydration is catalyzed by the H ions. The author detd. the hydration const. in more conc. solns. (0.0628-0.6573 g. HPO3 per g. soln.) and found that it increases regularly, from 0.00023 to 0.00062. Since the H+ conc. is less in conc. soln., these results are not in accord with the hypothesis of Montemartini and Egidi, unless the max. dissociation of HPO3 occurs at a conc. greater than that of a satd. soln. A second hypothesis is that the hydration is catalyzed by the HPO3 itself. This question is under investigation.

GEORGE W. MOREY.

The Preparation of Pure Potassium Ferrocyanide and its Analytical Control. KARL. Schröder. Wiesbaden. Z. anorg. Chem., 72, 89-99.—K<sub>4</sub>Fe(CN)<sub>6</sub>.3H<sub>2</sub>O crysts. from b. satd. soln. and when dried in the air between filter paper, shows a normal H<sub>2</sub>O con-

tent, detd. by drying to constant wt., care being taken to insure the smallest possible surface. As a check on its purity, Fe is detd. by the use of "cupferron," which the author recommends. K<sub>4</sub>Fe(CN)<sub>6</sub> can be titrated directly with KMnO<sub>6</sub> after decomp. with HgCl<sub>2</sub>. To 100 cc. cold satd. HgCl<sub>2</sub> soln. are added 2 g. K<sub>4</sub>Fe(CN)<sub>6</sub>.3H<sub>2</sub>O, dissolved in 50–100 cc. H<sub>2</sub>O. This resulting mixt. is added in small portions to 200 cc. satd. HgCl<sub>2</sub> heated to boiling. After the ppt. is dissolved the soln. is cooled, titrating mixt. added, and the FeCl<sub>2</sub> titrated with KMnO<sub>6</sub>. Results given are good. G. W. MOREY.

Potassium Barium Orthosulfantimoniate, (KBaSbS<sub>4</sub> + 6 H<sub>2</sub>O), (K<sub>2</sub>SbS<sub>4</sub> + Ba<sub>2</sub>Sb<sub>2</sub>-S<sub>8</sub> + 18 H<sub>2</sub>O). EMANUEL GLATZEL. Breslau. Z. anorg. Chem., 72, 100-5; see C. A., 5, 1883.—KBaSbS<sub>4</sub> + 6H<sub>2</sub>O, prepd. from Ba<sub>2</sub>Sb<sub>2</sub>S<sub>2</sub> and KCl, forms clear colorless or white crystals, which turn brown in the air. Very sol. in H<sub>2</sub>O soln., let stand in air becomes covered with brown crust, which is cleared up by KSH. Decomp. by acid with pptn. of SbS<sub>3</sub>, gives off H<sub>2</sub>O when heated and melts to a clear liquid, which solidifies to a cryst. mass.

George W. Morrey.

Equilibrium in the Precipitation of Lead Carbonate. W. Herz. Pharm. Inst., Univ. Breslau. Z. anorg. Chem., 72, 1006–8.—The author studies the equil. in the ppt. of PbCO<sub>2</sub> by shaking the Pb salt in excess with NaCO<sub>2</sub> soln. and titrating the Na<sub>2</sub>CO<sub>3</sub> in the supernatant liquid. With PbBr<sub>2</sub> the values 3.5 and 2.7  $\times$  10<sup>-4</sup> were obtained with 0.851 and 1.145 N Na<sub>2</sub>CO<sub>2</sub> for  $K = (\text{Na}_2\text{CO}_2)/(\text{NaBr})^3$ . With PbCl<sub>2</sub> the values obtained for  $K = (\text{Na}_2\text{CO}_2)/(\text{NaCl})^3$  were 1.2 and 1.0  $\times$  10<sup>-4</sup> with 1.145 and 1.85 N Na<sub>2</sub>CO<sub>2</sub> solns. With PbSO<sub>4</sub> the values 1.2, 1.0, 1.0, 1.0  $\times$  10<sup>-4</sup> were obtained; with Na<sub>2</sub>CO<sub>3</sub> solns. 0.2128, 0.4255, 0.6382 and 0.851 N. George W. Morey.

Constitution of Silver Ammonium Chloride. Jan Straub. Techn. Hochsch., Delft. Z. physik. Chem., 77, 331-8.—The author studies the AgCl-NH<sub>2</sub> equil. in strong NH<sub>2</sub> soln. with AgCl, 2AgCl.3NH<sub>2</sub>, and both compds. as solid phase. His results confirm those of Bodlander, the dissociated complex AgCl.2NH<sub>2</sub> being the only one present in soln. A. interprets his results by 2 methods, details of the calculations in both cases being given.

G. W. Morey.

Treatment of Wetable Sulfurs by Cholic and Tauric Acids. J. A. A. AUZIES. Bordeaux. Rev. gén. chim., 14, 278-80.—A very brief description of the synthesis of cholic and tauric acids and of the treatment of S to make it wetable by, H<sub>2</sub>O and metallic solns.

Chas. A. ROUILLER.

Calcium Carbonate. W. MEIGEN. Freiburg i/B. Chem. Ztg., 34, 1015.—
Aragonite powder boiled with Co(NO<sub>2</sub>)<sub>2</sub> soln. gives a lilac coloration, calcite gives a light blue. Aragonite powder, with FeSO<sub>4</sub>, gives a dark green ppt.; calcite gives a yellow ppt. With other metals the difference in rate of action can only be detd. quantitatively. The reaction rate is greatest for aragonite with Zn, Mn, Co and Fe, and for calcite Cu, Pb and Ag. This difference in reaction velocity can only be explained on the basis of mol. differences besides the crystallographic ones. The ppts. obtained from Ca salts and alk. carbonates were identified by this method; as stated by Rose, calcite was formed in the cold dil. and aragonite in hot dil. solns. From conc. solns. it is pptd. in the amorphous condition; in the hot it rapidly crystallizes; in the cold it remains amorphous a long time, gradually forming spheroidal crystals which qualitatively are like aragonite but quantitatively their reaction velocity is different from both. The d. is different (2.56). Its properties coincide with those of the 3rd modification of CaCO<sub>2</sub> described by Vater and it has been called "Vaterite."

E. J. WITZEMANN.

The System: Sulfur-Tellurium (CHIKASHIGE). 9. Catalytic Properties of Asbestos (CHRISHVILI). 7.



Electrical Conductivity of Diamonds (DOELTER). 8.

FRIEDERICI, K.: Die Einwirkung von Wasserstoffsuperoxyd auf die Alkalisalze der Molybdänsäure. Berlin: E. Ebering. 8°, 1.30 M.

KIPPING, F. S. AND PERKIN, W. H.: Inorganic Chemistry. (Two parts.) London: 8°, \$3.00.

MAXTED, E. B.: Ueber die Nitride von Eisen, Nickel und Kobalt. Ueber das Bleicoulometer. Berlin: E. Ebering. 1 M.

# 7. ANALYTICAL CHEMISTRY.

#### E. G. R. ARDAGH.

Electroanalysis. Report of Committee read before British Association, Portsmouth, 1911. Chem. News, 104, 141.—Attention is drawn to the work done on the application of the electrometric method to the titration of weak acids in such liquids as tan liquors. Detn. of the end point is discussed. F. M. Perkin has designed an app. (an improvement on E. F. Smith's) of fused quartz for the electrolysis of alkali salts, by means of which both base and acid can be detd.

E. G. R. Ardagh.

Rapid Qualitative Test for the Elements whose Sulfides are Precipitated by Hydrogen Sulfide in Acid Solution. E. Pozzi-Escot. Bull. soc. chim., 9, 812-4.—Attack the pptd. sulfides with HCl (1-4). Residue (1) contains Cu, Mo, As, Bi, Hg, Au, Pt; filtrate (2) contains Pb, Sn, Sb, Cd, Zn. Treat (1) with HNO<sub>2</sub> to get a residue (3) containing Hg, Au, Pt, and a filtrate (4) containing Cu, Mo, As, Bi. Dissolve (3) in aqua regia, replace HNO<sub>2</sub> by HCl. Detect Hg by SnCl<sub>2</sub> or KI, Au by FeSO<sub>4</sub>, and Pt by KI. In (4) detect Cu by NH<sub>4</sub>OH or Fe(nail), Mo by tannin, As by AsH<sub>2</sub> with Zn, Bi by b. 1 or 2 min. with conc. HCl and SnCl<sub>2</sub> in large excess. Treat (2) with NH<sub>4</sub>OH to get a residue (5) containing Pb, Sb, Sn, and a filtrate (6) containing Cd, Zn. Dissolve (5) in HCl and detect Pb by H<sub>2</sub>SO<sub>4</sub>, Sb by adding KOH in excess, then AgNO<sub>2</sub> and then an excess of NH<sub>4</sub>OH, which gives a black ppt. if Sb is present; Sn by pptg. on Zn and detecting SnCl<sub>2</sub> by HgCl<sub>2</sub>. In (6) detect Cd by H<sub>2</sub>S, Zn by H<sub>2</sub>S + NH<sub>4</sub>OAc in the filtrate from CdS. Rapidity is claimed.

Determination of Ferric Oxide in the Presence of Alumina. KRIEGER. Chem. Ztg., 35, 1054.—Place the ignited  $Fe_2O_3$ ,  $Al_2O_3$  ppt., without previously pulverizing, in an Erlenmeyer, add 25 cc. conc.  $H_2SO_4$  and some water, heat to b. for 5 to 15 min., dil. with 50 cc.  $H_2O_7$  reduce with Zn and titrate with KMnO<sub>4</sub>. No results are given but the  $Fe_2O_3$  is said to dissolve, leaving the  $Al_2O_3$  almost unattacked. E. G. R. A.

New Reaction for the Detection of Hydrogen Peroxide. O. v. Sobbe. Chem. Zig., 35, 898.—A portion of a soln. containing 3 drops 3% H<sub>2</sub>O<sub>2</sub> in 100 cc., produced in ammoniacal AgNO<sub>3</sub> soln. a characteristic gray ppt. insol. in HCl. Five drops of a similar H<sub>2</sub>O<sub>2</sub> soln. were required to produce a blue color with KI-starch soln. or ZnCl<sub>2</sub>-starch-soln, the latter soln. being more stable.

William Blum.

A New Reaction for the Detection of Hydrogen Peroxide. B. M. MARGOSCHES. Chem. Ztg., 35, 955.—Von Sobbe's use of ammoniacal AgNO<sub>2</sub> to detect H<sub>2</sub>O<sub>2</sub> (preceding abstr.) is not new, but is substantially the same as recommended by Birckenbach in 1909 and by Böttger in 1871.

E. G. R. ARDAGH.

The Separation of Iron and Vanadium by the Ether Method. E. Deiss and H. Leysaht. Kgl. Materialprüfungsamt Grosslichterfelde West. *Chem. Zig.*, 35, 878–9; cf. C. A., 5, 3390.—To the Fe-V soln. contained in the special separatory funnel is added HCl (d. 1.19) satd. with Et<sub>2</sub>O, 6 cc. for each g. Fe, keeping the soln. cool.

After shaking, the app. is let stand until the liquid seps. into 2 layers, the lower of which is drawn off into the lower bulb of the separatory funnel. The HCl soln. in the stop-cock bore is washed out with HCl (d. 1.10) satd. with Et<sub>2</sub>O, after which the small amt. of V contained with the Fe in the V<sup>V</sup> state is removed by adding a few drops of H<sub>2</sub>O<sub>8</sub> and 10 cc. HCl (1.10) satd. with Et<sub>2</sub>O, adding this to the main portion. The Fe carried through with the V is sepd. by shaking with a second portion of Et<sub>2</sub>O satd. HCl (d. 1.19). The HCl soln. containing the V is then run into an evapg. dish, V being detd. in the ordinary manner. The Et<sub>2</sub>O soln. should be shaken several times with HCl, after addition of a few drops H<sub>2</sub>O<sub>2</sub>, to remove all V. Fe can be detd. in the Et<sub>2</sub>O soln. Results given show errors of 0.1-0.3 mg., using Fe-V mixts. corresponding to 3.9-0.05% V.

The Treatment of Insoluble Residues. E. EBLER. Heidelberg. Z. anal. Chem., 50, 610-5.

Percy H. Walker.

A Modified Procedure for the Detection of Silicates, Fluorides and Fluosilicates. P. E. Browning. Kent Chem. Lab., Yale Univ. Am. J. Sci., 32, 249-50.—The work was undertaken to devize a method by means of which the test for SiF<sub>4</sub> could be made more delicate and trustworthy. When moist black paper is brought in contact with the fumes of SiF<sub>4</sub>, the deposit of Si(OH)<sub>4</sub> is very easily detected. A small Pb cup about 1 cm. in diam. and depth, with a flat piece of Pb with a hole in the center as cover was used. About 0.1 g. of CaF<sub>2</sub> along with the silicate was placed in the cup, and the mixt. covered with a few drops conc. H<sub>2</sub>SO<sub>4</sub>. On the upper side of the cover a piece of moist black filter paper was placed, and the cup was then heated for 10 min. on a steam bath. This method will serve to detect 0.1 mg. of Si or F.

A. V. DE LAPORTE.

The Qualitative Detection of Certain Elements which Form Insoluble Sulfates. Ba, Sr, (Ca) and Pb. P. E. Browning and P. L. Blumenthal. Kent Chem. Lab., Yale Univ. Am. J. Sci., 32, 246-8.—The sources of error in testing for the alkali earth elements in the ordinary course of analysis are described. The authors suggest the following method: About 10 cc. of a soln. of Hg', Ag, Pb, Ba, Sr, Ca, and other elements are treated with HCl in faint excess, the pptd. chlorides filtered off, and about 5 g. of (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> added to complete pptn. After gently warming, the sulfates are filtered and washed with a satd. soln. of AcONH, until the washings give no test for Pb with H.S. The filtrate and the washings are treated as in the ordinary course of analysis. To the pptd. sulfates a small amt. of pure sugar C is added and the paper rolled up and ignited to redness for a few min. either in a closed glass tube or in a covered crucible. The fused mass is treated with about 5 cc. of 50% AcOH and warmed, the mixt. filtered and the residue washed with 5 cc. H<sub>2</sub>O. BaCrO<sub>4</sub> is pptd. with K,CrO, and filtered. The Ca and Sr are pptd. as carbonates by b. the filtrate with Na<sub>2</sub>CO<sub>2</sub>. If the ppt. is small it can be tested spectroscopically. If not too small dissolve in HNO, after washing, and the Sr and Ca can be sepd. by dehydration with amyl alc. This sepn. may, with advantage, precede the group pptn. by H<sub>2</sub>S in ordinary analysis. A. V. DE LAPORTE.

Potassium, its Qualitative Detection as the Cobaltinitrite. L. T. BOWSER. J. Am. Chem. Soc., 33, 1566-9.—This is a modified colorimetric method; the cobaltinitrite reagent is used in presence of EtOH, and is said to give a reaction when K is present to the extent of 2 parts in a million. It is essential that no NH, salts be present, and that even the atm. be as free as possible from NH, fumes, since these latter ppt. with the reagent.

L. J. ROGERS.

Alkalinity of Magnesium Ammonium Phosphate and Acidimetry of Ammonium Phosphomolybdate. III. F. HUNDESHAGEN. Stuttgart. Z. öffent. Chem., 17,

322-4.—Comparative analyses of raw phosphate from 'various sources are given, showing a close agreement between H.'s volumetric methods and the citrate method. When  $H_2SO_4$  is used in analyzing Thomas meal the vol. of insol. sulfate introduces an error when the soln is made up to vol. accounting for the high results obtained using this method. The citrate method used with the titration of the ppt. is worthy of careful trial in analyzing phosphates.

D. S. Pratt.

Catalytic Properties of Asbestos. P. I. CHEISHVILI. J. Russ. Phys. Chem. Soc., 42, 856-62.—Asbestos, even when purified by digestion with acids and calcination, reduces an acid soln. of KMnO<sub>4</sub> up to the eighth filtration. A filter containing about 1 g. asbestos reduces 0.025 cc. of 0.05 N KMnO<sub>4</sub> in each of the first 7 filtrations, when 10 cc. of the liquid passes through it. The slight ppt. formed on the asbestos cannot be MnO<sub>2</sub>, since the amt. of KMnO<sub>4</sub> reduced is constant, though the amt. of the ppt. gradually increases. In filtering KMnO<sub>4</sub> soln. it is preferable to use asbestos and make the above correction, than to use glass wool which never gives a clear filtrate.

H. M. GORDIN.

The Indicator of Rupp and Loose. I. I. OSTROMISSLENSKII AND I. S. BABADZHAN. Lab. Org. and Gen. Chem. Imper. Techn. School, Moscow. J. Russ. Phys. Chem. Soc., 42, 609-11.—Not succeeding in obtaining the indicator of Rupp and Loose by following their directions (C. A., 3, 523), the authors ask them for more explicit details.

H. M. GORDIN.

The Analysis of Monazite Sand. G. CHESNEAU. Compt. rend., 153, 429-31.-After fine grinding, 2.5 g. of the mineral is fused with 15 g. KNaCO, the melt dissolved in b. 1% Na, CO, soln., filtered, and washed with cold H.O. The residue is treated with 5% HCl and the fusion repeated on the residue. The residue, containing TiO, ZrO<sub>2</sub>, and traces of SiO<sub>2</sub> and CaO, and the NaCl soln. containing Al<sub>2</sub>O<sub>2</sub>, SiO<sub>2</sub> and H<sub>2</sub>PO<sub>4</sub> are treated in the usual manner. The HCl soln. containing rare earths, TiO<sub>2</sub>, ZrO<sub>3</sub>, Fe, etc., is made exactly 4% acid with HCl, 4 g. oxalic acid crystals added for every 100 cc. soln., let stand 2 hrs. and then filtered. The filtrate, containing TiO<sub>2</sub>, ZrO<sub>3</sub>, Fe, etc., is treated in the usual manner. The oxalates are washed with oxalic acid soln., dried, and ignited to the oxides. These are taken up with conc. HNO, with the aid of heat, using H<sub>2</sub>O<sub>2</sub> to assist in soln. if necessary. The clear soln. is evapd., the residue dried at 130°, dissolved on 150 cc. H<sub>2</sub>O, and Th sepd. by the H<sub>2</sub>O, method, making a double pptn. The combined filtrates are diluted to 500 cc., the rare earths pptd. from all but 100 cc. with NH<sub>4</sub>OH, and weighed. Ce is detd. in the 100 cc. portion volumetrically, using Job's PbO, method. The Y earths are detd. by the double K<sub>2</sub>SO<sub>4</sub> method, La, Pr and Nd together by difference. Analysis of a Madagascar monazite GEORGE W. MOREY. sand is given.

The Use of Sulfur Monochloride in the Determination and Analysis of the Rare Earth Minerals. W. B. Hicks. Univ. Penn. J. Am. Chem. Soc., 33, 1492-6.—H. uses S<sub>2</sub>Cl<sub>2</sub> for opening up rare earth minerals containing Cb, Ta, Ti, etc. The decomp. is complete and easy, and the earth acids are completely sepd. from the rare earth.

George W. Morry.

Precipitation of Iron with Hydrazine Hydrate. E. Schirm. Chem. Ztg., 35, 897.—S. found that upon the addition of N<sub>2</sub>H<sub>4</sub>OH to Fe<sup>III</sup> salts a red flocculent ppt. of Fe(OH)<sub>2</sub> is produced. On b. this ppt. becomes black and granular, and the filtrate is free from Fe. When pptd. in the presence of sulfates the ppt. is free from SO<sub>4</sub>. For the quant. sepn. of Fe and SO<sub>4</sub> 2 methods may be used. In t case the b. soln., containing Fe as Fe<sup>III</sup>, is nearly neutral with NH<sub>4</sub>OH, pptd. with N<sub>2</sub>H<sub>4</sub>OH in slight excess, the ppt. filtered out, washed with hot H<sub>2</sub>O, ignited and weighed as Fe<sub>2</sub>O<sub>2</sub>. In the filtrate SO<sub>4</sub> may be detd. as usual. To reduce the consumption of the expensive

N<sub>2</sub>H<sub>4</sub>OH, the soln. may be made strongly alk. with NH<sub>4</sub>OH, and a few drops of N<sub>2</sub>H<sub>3</sub>OH added, producing the same black granular ppt. The results obtained by both methods were good. Attempts to sep. Fe<sup>III</sup> and Zn by this method were unsuccessful.

WILLIAM BLUM.

Precipitation of Aluminium, Chromium and Iron with Ammonium Nitrite. E. Schirm. Chem. Zig., 35, 979-80.—S. attempted to sep. Al, Cr and Fe from Mn and from Zn by pptn. with NH<sub>4</sub>NO<sub>2</sub> as previously described (C. A., 3, 2915). The results for Mn were always about 2% low after a single pptn. of Al, Cr or Fe. Results for Zn were 20% low, due to pptn. of the Zn as aluminate, chromite or ferrite. The use of nitrites of quaternary organic NH<sub>4</sub> bases, such as trimethylphenylammonium nitrite, instead of NH<sub>4</sub>NO<sub>2</sub> when excess NH<sub>4</sub> salts are present, was suggested. W. B.

The Assay of Wolframite Concentrate. H. W. HUTCHIN. Analyst, 36, 398-403.—The following 4 methods for the detn. of W are given: (1) Aqua regia-NH4 tungstate method: To 1 g. finely powdered wolframite add about 10 cc. conc. HCl and agitate to prevent caking. Add more acid (about 120 cc. in all), cover and evap. to 5 cc. Allow to cool, add 5 cc. strong HNO2 and digest at nearly a b. temp. for 5-10 min. Dil. to about 100 cc., allow to settle, filter and wash with H<sub>2</sub>O, working so as to obtain as little H<sub>2</sub>WO<sub>4</sub> on the paper as possible. To the filtrate add 10 cc. dist. H<sub>2</sub>O and 10 cc. NH<sub>2</sub>OH (1:1) in small quantities down the sides of the beaker. Heat to b. and stir well (if the suspended matter does not readily subside b. until it does). Allow to settle and while still hot filter through the original paper. After washing, evap. the soln. of NH, tungstate in the filtrate to a small vol., transfer to a weighed Pt dish, dry on the water bath and ignite. Weigh as WO<sub>3</sub>. To the heavy residue add 5 cc. HCl, cover with the original watch glass, b. till nearly dry, add a few cc. of HNO, and heat again for 10 min. Dil. with H<sub>2</sub>O, let stand till the deposit has subsided, filter and wash. Wash the filter paper (also the original filter paper) with a little hot dil. NaOH soln. followed by dist. H<sub>2</sub>O and collect the alk. liquors. If the watch glass is stained with the WO, dissolve with a little dil. NaOH and add to the alk. liquors. Heat to b., add about 2 g. NH<sub>4</sub>NO, and stir while b. Filter through 1 of the papers previously used and wash. Nearly neutralize the filtrate with dil. HNO, add few drops of HgNO<sub>2</sub> soln., stir well, filter, wash, dry, ignite and weigh as WO<sub>2</sub>. Add this wt. to the wt. of the WO, in the Pt dish. The residue may contain as high as 2% of WO. (2) Aqua regia-HgNO, method: Proceed as in (1) through first filtration. To this filtrate add 15 cc. dist. H<sub>2</sub>O and 5 cc. of 10% NaOH soln., wash cover glass with dil. NaOH, run a few cc. of the NaOH through the filter, wash with H<sub>2</sub>O, collect filtrate and washings, heat to b., add 3 g. NH<sub>4</sub>NO<sub>2</sub> and b. for a few m. Allow to settle, filter and wash with weak NH4NO2 soln. Dil. to about 200 cc., acidify with HNO2, make faintly alk. with dil. NH4OH. Add sufficient HgNO2 soln., neutralize with pptd. HgO, stir well, filter and wash by decantation. Transfer ppt. to paper, complete the washing, dry, ignite and weigh as WO<sub>2</sub>. The residues should be reheated as in (1). (3) Soda digestion method: Digest 0.625 g. sample with 20 cc. of 25% NaOH on a water bath for 45 min. Stir well at first to prevent caking. Dil. the ext. with H<sub>2</sub>O, add a little Na<sub>2</sub>O<sub>2</sub>, transfer to a 250 cc. flask, make to the mark, take a 200 cc. aliquot, representing 0.5 g. and treat like the filtrate in (2), beginning with the acidification with HNO<sub>2</sub>. (4) Soda fusion-HgNO<sub>3</sub> method: Fuse 0.625 g. finely powdered sample in a Ni dish with about 1 in. of stick soda and 3 g. Na<sub>2</sub>O<sub>2</sub> to a clear melt. Pour the molten charge onto sheet of Ni and when cold transfer to a beaker and dissolve in H<sub>2</sub>O. If soln. is green add Na<sub>2</sub>O<sub>2</sub>, transfer to 250 cc. flask, make to vol., take an aliquot of 200 cc. and proceed as in (3). All 4 methods give good results but H. prefers (1), because it may be worked with 1-2.5 g. of sample while in the other methods good results are obtained only on about 0.5 g. C. P. WILSON.

Determination of Gold and Silver in Copper. E. F. KERN AND A. A. HEIMROD. School of Mines, Columbia Univ., N. Y. Met. Chem. Eng., 9, 496-9.—After describing several partially successful methods, results from which are also recorded, the authors give in detail the methods finally adopted. For Gold: Standard Au alloys were made and used. Place 1 A. T. in a 900 cc. beaker, add 20 cc. H.O, cover, and add 90-100 cc. HNO<sub>2</sub> (1.42) in quantities of 20-25 cc. at a time. When dissolved, add 40 cc. H<sub>2</sub>SO<sub>4</sub> (1.84), stir to break up CuSO<sub>4</sub> crystals, and evap. on a sand bath to SO<sub>5</sub> fumes. Cool, add 500 cc. H<sub>2</sub>O, warm till dissolved, add 10 g. FeSO<sub>4</sub>.7H<sub>2</sub>O dissolved in H<sub>2</sub>O, 25 cc. of a satd. soln. of NaCl, and enough AgNO, to make a bead of about 8 times the weight of the Au present. Add the AgNO, to the hot soln. and stir to coagulate the AgCl which carries down the finely divided Au. Filter, wash out Cu salts, place filter and contents on a litharge-glazed scorifier with about 20 g. test-Pb, burn off paper carefully, add 20 g. more test-Pb and a pinch of borax, scorify, cupel on a medium hard bone-ash cupel, part with 1-4 HNO, wash, anneal and weigh. Results given of check assays agree excellently. For Silver: Standard Ag alloys were made. Weigh out 5, 10, or 20 g., depending on the Ag content, add conc. HNO (3 cc. per g.), cover beaker, evap. till Cu(NO<sub>2</sub>), begins to cryst. This ensures all NO<sub>2</sub> being driven off (important). Add 400 cc. cold H<sub>2</sub>O and 25 cc. satd. NH<sub>4</sub>CNS (25 g. per l.) as quickly as possible with const. stirring to the cold soln. Filter and wash out Cu salts. Place filter and ppt. in the beaker in which pptn. was made, digest on the hot plate with 80 cc. conc. HNO, till the paper is decomposed and the ppt. dissolved. Evap. till all NO, is driven off, but do not carry too far or brown organic matter will form which obscures the end point of the titration. Cool, add 300-350 cc. H<sub>2</sub>O, and 20 cc. satd. Ba(NO<sub>3</sub>)<sub>2</sub> to ppt. SO<sub>4</sub>--. Add 3 cc. satd. Fe-alum, and titrate at ordinary temp. with NH<sub>4</sub>CNS (3.2 g. per l.), standardized against electrolytic Ag, to a faint orange. Results recorded agree excellently. E. G. R. ARDAGH.

Assay of Used Plumbago Crucibles for Gold. John Watson. J. Chem. Met. Min. S. Africa, 12, 13; cf. C. A., 5, 2608.—Discussion. Mr. Dan Nicholas gives this method. He regards 0.1 or 0.2 A. T. too small an amount to take. He uses 0.5 A. T. with excess of flux. (The flux is made of soda 7, borax 4, and PbO 1½ parts.) SiO<sub>2</sub> may be used if necessary. Fuse about ½ hr. in an ordinary crucible. Pour off the fluid slag temporarily, add niter to oxidize the unfused material remaining, rotating or shaking to assist the operation, return the slag to the crucible, replace it in the fire, and complete the fusion. Charcoal may be added to make sure of getting a Pb button. Ag may be added to the Pb button in amts. required for parting purposes.

Analysis of Zinc Dust. Anon. *Mining Sci. Press*, 103, 356.—A description is given of the Drewson method which depends upon the fact that Zn dust will reduce a soln. of  $CrO_3$  without evolution of H. To the Zn is added, in slight excess, an acidified stand. soln. of  $K_2Cr_3O_7$ , and the excess is then titrated back with standard FeSO<sub>4</sub>. The method is recommended as both convenient and reliable. EDW. WOLESENSKY.

Assay of Tin. Ernest A. Lewis. London Min. J., 1911, 606; through J. Chem. Met. Min. S. Africa, 12, 32-3.—Volumetric assays tend to give low results, on account of the ready oxidation of Sn". The electrolytic method is much more accurate. A weighed amt. of the ore should be cleaned by digestion in HNO<sub>2</sub>. The material is then dried and reduced in a combustion tube by heating in a current of H or of coal gas. Dissolve in HCl and oxidize to Sn<sup>IV</sup> by addiding a few drops of HNO<sub>2</sub>. Dil., filter, and take an aliquot portion containing about 0.4 g. Sn. Add NH<sub>4</sub>OH cautiously until a permanent ppt. is obtained. Redissolve by adding a hot soln. of 3 g. NH<sub>4</sub>HC<sub>2</sub>O<sub>4</sub> and 7 g. H<sub>2</sub>C<sub>2</sub>O<sub>4</sub>. Heat to b. and pass in H<sub>2</sub>S for an hr. Filter and wash with as little H<sub>2</sub>S water as possible. Boil the soln. to remove H<sub>2</sub>S completely (2 hrs.)

and then cool and electrolyze. [Apparently the author used rotating electrodes.—ABSTR.] The only likely contaminant is Fe. After weighing up, the metal can be dissolved in HCl, and if Fe is found it may be detd. and deducted. E. WALLER.

Combustion under Pressure for Determining Carbon in Steel. P. MAHLER AND E. GOUTAL. Compt. rend., 153, 549-51.—The bomb employed has a capacity of 1 l. and is not lined with enamel. When desired one can insert a porcelain vessel. The charge is placed in a refractory capsule and is fired electrically by means of a very fine Fe wire. Charge of steel 2 to 5 g. Pressure of O 5 to 8 atm. The gas is passed through absorbers containing Ba(OH)<sub>2</sub> soln. which is titrated with oxalic acid (10.5 g. per l. 1 cc. — 1 mg. C) using phenolph. The results recorded agree fairly well with the works analyses for low C steels, but for the most part are all considerably higher for special steels.

E. G. R. Ardagh.

Carbon Determination in Steels and Irons in the Electric Furnace. H. Augustin. Z. angew. Chem., 24, 1800-3. — The method originated with Richard Lorenz (Ibid., 6, 313, 395, 411 (1893)). The author's modification makes it suitable for technical practice. From 1-15 g. of turnings are used, depending on the C content and degree of accuracy desired. The Lorenz method of covering or mixing the sample with PbCrO4 is not necessary, but one cannot substitute Pt as contact substance for the CuO since with rapid combustion the O may be almost completely consumed. The furnace (illustrations) contains 4 porcelain tubes to permit 4 samples being burnt simultaneously. The tubes are glazed inside and out and have an inside diam. of 16-18 mm. A thin walled muffle which surrounds the tubes is wound with 2 independent coils of Pt wire. One resistance heats the combustion ends of the tubes to 1000°, the other the CuO and PbCrO4 ends to 800°. Two thermoelements placed in the central space between the 4 tubes are connected with a galvanometer.

E. G. R. ARDAGH.

New Method for the Rapid Determination of the Total Carbon in Iron, Steel and Ferro Alloys. A. L. Queneau. Met. Chem. Eng., 9, 441-2.—The sample is burned in an ignited asbestos boat in an atm. of O, the combustion being started by means of a small arc flashed through the steel sample and the resulting CO2 absorbed in NaOH (4 g. per 1.) and detd. The combustion is made in a 1. flask closed by a rubber stopper (protected by asbestos), through which pass 2 Cu electrodes and a Cu tube. To the Cu tube is attached a Cu wire, supporting the boat and a basket of Cu gauze under the boat to catch any material that may escape. The Cu tube terminates 5-10 mm. above the boat and serves for introducing O. There is a Hg trap to prevent any CO, passing back. Thin, voluminous drillings are desired, avoiding dust and needle-like particles. Hard or tempered steels are crushed and passed through 100 mesh, pig irons through 80 mesh, ferro-alloys through 120 mesh. For soft steels a good combustion is readily obtained without the addition of any oxidizing agents; for all other metals it is necessary to add PbO<sub>2</sub> or Bi<sub>2</sub>O<sub>4</sub>. With all materials containing over 1.5% C it is necessary to add fine drillings from an extra soft and pure steel (0.05-0.07 C), making a correction for same. For steels with less than 1.5% C use a 1 or 2 g. sample and 0.1-0.2 g. PbO, for hard steels or steels with over 1.5% C use 1 g. with 0.2 g. PbO<sub>3</sub>; pig iron, ferrochromes with 1.5% C, ferrosilicons 10-90% use 0.5 g. with 0.2 g. PbO<sub>2</sub> and 1 g. soft steel; ferrochromes with 6-10% C use 0.25 g. with 0.2 g. PbO<sub>2</sub> and 2 g. soft steel: ferromanganese 0.25 g. with 0.2 g. PbO, and 1 g. soft steel or 0.5 g. with 0.2 g. PbO<sub>2</sub> and 2 g. soft steel; ferromolybdenum use 0.5 g. with 0.5 g. PbO<sub>2</sub> and I g. soft steel (with less PbO, some MoO, is volatilized and is absorbed by the NaOH); ferrotungsten use 0.5 g. with 0.1 g. PbO, and 1 g. soft steel (with more than o.1 g. PbO, the combustion is too intense and the boat is bored). When soft steel is used, these drillings are spread on the bottom of the boat and the mixt. of sample

and PbO<sub>2</sub> placed on top. Sulfates and phosphates remain in the boat, from which they may be collected and detns. made. As soon as the combustion has begun the current is cut off, the metal continuing to burn until combustion is complete. After combustion, the connection to the O is closed, the flask disconnected and cooled by plunging into water; then open quickly, remove stopper with boat, plain stopper is put in, shaken 10 sec. and titrated. A detailed drawing of the app. is given. The method is as accurate as the standard combustion method, and a detn. may be made in from 5-7 min. The accuracy is independent of the presence of Ni, Cr, W, Mo, V, Ti, S or P, and even gray Fe high in graphite may be completely burned. No results are given.

The Determination of Manganese in Vanadium and Chrome Vanadium Steels. J. R. CAIN. J. Ind. Eng. Chem., 3, 630-1—Dissolve the steel in  $H_2SO_4$  (1: 10) and sep. Cr and V by pptn. with  $CaCO_6$  (see C. A., 5, 2791). Det. Mn in the filtrate by the bismuthate method.

Cupferron: Its Use in Quantitative Analysis. O. BAUDISCH AND V. L. KING. J. Ind. Eng. Chem., 3, 629-30.—The advantages of pptg. Fe with "cupferron" and the method of using it are set forth. The method of prep. is given in detail.

E. G. R. ARDAGH.

Quantitative Separation with Cupferron. O. BAUDISCH. Chem. Ztg., 35, 913.—
"Cupferron" is  $C_0H_0(NO)ONH_0$ . Unlike the free nitrosophenythydroxylamine it is not explosive and can be kept indefinitly by simply adding a lump of  $(NH_0)_0CO_0$  to the container. An ammoniacal aq. soln. of cupferron will keep for a month. The method of prep. is given. (See preceding abstr.)

E. G. R. Ardagh.

A Rapid and Accurate Method for the Analysis of White Metals. J. C. BENEKER. J. Ind. Eng. Chem., 3, 637-8.—B. describes a method of analysis of alloys containing Sn, Sb, Cu and Pb.

PERCY H. WALKER.

The Volumetric Chromate Determination of Lead. J. WADDELL. J Ind. Eng. Chem., 3, 638-40.—The method described depends upon the pptn. of Pb as PbCrO<sub>4</sub> which is used to set I free from KI; this I is titrated with Na<sub>2</sub>S<sub>4</sub>O<sub>5</sub>.

P. H. W.

Notes on Technical Analysis. R. S. DAVIS. Met. Chem. Eng., 9, 458-9.—Wulfenite ores are analyzed by decomposing with HCl and HNO, and after filtration sepg. Pb from Si by soln. in AcONH, and AcOH and estg. either by titration with NH. molybdate or gravimetrically as PbSO4. Mo is pptd. from the filtrate by H<sub>2</sub>S and weighed as MoO<sub>1</sub>. If much Mo is present a further treatment of the pptd. MoS<sub>2</sub> is necessary, the Mo being weighed as PbMoO<sub>4</sub>. The estn. of Mo by the Zn reduction process is accurate only when little Mo is present. V is detd. in the filtrate from the MoS, by KMnO. W and Si are detd. in a sep. sample. The ore is treated with HCl, evapd. to dryness, treated with HCl, and SiO, and WO, filtered and washed by decantation in the hot, dil. HCl till free from PbCl<sub>2</sub>. WO<sub>2</sub> is dissolved out with NH<sub>4</sub>OH and the soln. is evapd. and the residue ignited and weighed. Any SiO, is removed by HF and the residue is again weighed. The residue from the NH4OH treatment plus any found in the WO, gives the total SiO. The following method for the analysis of ferroboron is recommended for accuracy and rapidity. The powdered sample is fused with KNO, and Na, CO,. Mn is pptd. by H,O, from the soln. of the fusion. After acidifying, adding CaCO<sub>2</sub>, b. and filtering, phenolph. is added and o.1 N NaOH is run in to a pink color. Manitol is added and NaOH to restore the pink color. A q.5 g. steel sample containing 4% Mn and 0.03 g. B gave by this method 0.0299 g. B. Mn in the residue from the fusion is detd. by the bismuthate method, Si in the usual way, and C by direct ignition in O, the sample being spread over ignited asbestos in the combustion boat. M. C. Boswell.

Contamination of Laboratory Samples by Iron Derived from Crushing Machinery.

G. A. James. Chem. Eng., 14, 380-1.—J. calls attention to the very large amt. of Fe frequently introduced into finely ground samples, but offers no satisfactory method of preparing such samples.

Percy H. Walker.

Unavoidable Errors in Sampling. Morton Webber. Mining Sci. Press, 102, 846.—Unavoidable errors in mine valuation, frequently in favor of the mine, may result in various ways. Where the vein is thin and the surrounding rock soft much waste finds its way into the mill along with the ore, but may not be considered in selecting the sample. Where filling is necessary, or in cases of underground sorting, appreciable quantities of ore are sometimes lost. Again, where the ore consists of two or more minerals of different degrees of hardness, the softer and more friable are likely to be abnormally represented in the sample.

EDW. WOLESENSKY.

Extraction of Gas from Copper by Chemical Reaction and the Determination of Oxygen. M. Guichard. Compt. rend., 153, 272-5; cf. C. A., 5, 3384.—If, in a vacuum at high temp., Cu is converted to Cu<sub>2</sub>I<sub>2</sub> by subliming pure dry I over it, a small quantity of gas is released (22 cc. per 100 g. Cu in the expt. carried out). Heating Cu in an insufficient amt. of pure O (C. A., 5, 2044) yields a small quantity of unabsorbable gas (in 1 case 30 cc. from 100 g. Cu). The error in the detn. of O by means of electrolytic Cu varies from 6 in 1000 down to 3 in 10,000, according to the duration of the heating of the Cu in a vacuum before admitting the O. The latter degree of accuracy was attained by previously heating for 184 hrs. at 600°.

D. M. LICHTY.

Contribution to the Valuation of Calcium Nitrite and Nitrate. STUTZER AND GOV. Chem. Ztg., 35, 891.—If nitrite is estd. by oxidizing with KMnO<sub>4</sub> in presence of a sufficiently restricted quantity of H<sub>2</sub>SO<sub>6</sub>, the total nitrate may then be detd. by pptn. with nitron (cf. M. Busch, Ber., 38, 861; 39, 1401). Neither oxidation with H<sub>2</sub>O<sub>2</sub> nor destruction of the nitrite with hydrazine sulfate lead to a correct estn. of nitrate as nitron salt. Because of the expensiveness of nitron, the authors prefer to det. total N by the method of Devarda. Good results are obtained. Treat 50 cc. of the soln. (10 g. nitrate or nitrite in 1 l. H<sub>2</sub>O) with 50 cc. H<sub>2</sub>O, 50 cc. of 33% NaOH soln., and 2.5 g. of Devarda metal (50% Cu, 45% Al, 5% Sn). After the evolution of H subsides, the mixt. is very gradually warmed and, after 1 hr., the NH<sub>2</sub> is completely distilled off and estd. For titrating the nitrite, 0.2 N KMnO<sub>4</sub> or H<sub>2</sub>C<sub>2</sub>O<sub>6</sub> acidulated with dil. H<sub>2</sub>SO<sub>6</sub> is used and warmed to about 50°.

New Method of Simultaneous Determination of the Various Elements of an Organic Substance. Analysis of a Substance Containing C, H, O and N. J. A. A. Auzies. Bull. soc. chim., 9, 814-8.—The novelty consists in the use of thorinated asbestos or cotton instead of CuO in the combustion tube. The thorinated material is prepared by satg. the vehicle with a soln. of a Th salt, dipping it in NH<sub>4</sub>OH and igniting. The products of combustion are passed first through a weighed tube of CaCl, to det. H, then a titrated HCl soln. of Cu<sub>2</sub>Cl<sub>2</sub> for N (Ferd. Jean process), then weighed KOH bulbs for C. The Th compd. converts N into NO, which is titrated in the Cu<sub>2</sub>Cl<sub>2</sub> soln, with standard SnCl<sub>2</sub>. Analysis of a Substance Containing C, H, O, N and S. Ibid., 815.— Thorinated cotton is used and the SO, is caught in a plug of PbO. The charge should be sepd. from this by a wad of asbestos, or better by using 2 of the specially designed porcelain "boats," I for the charge, the 2nd for the PbO<sub>2</sub>. After the combustion the unattacked PbO<sub>a</sub> is dissolved out by HCl, leaving PbSO<sub>a</sub> to be weighed up. The "boats" are really tubes with a shoulder at one end, which will just slide into the combustion tubes. Length internally 80 mm., diam. 13 mm.—of end apertures 7 and 9 mm., resp. Analysis of a Substance Containing C, H, N, O, Cl, Br and I. Ibid., 815-9.—Ag, CrO, is added to the above arrangement for absorbing the halogens. It is not stated where this is interposed in the train. After the combustion, AgCl and the surplus Ag.CrO. are dissolved off by NH<sub>4</sub>OH and the residual AgBr and AgI sepd. by dissolving out the latter by KCN. The AgCl is sepd. by pptg. with AcOH and dissolving in KCN for detn. The results of 3 sep. analyses of a mixt. of organic substances so mingled as to contain all the elements enumerated, show a very good agreement. O is detd. by difference. The solns. for the Jean method of detg. NO<sub>2</sub> are prepared by dissolving 35 to 40 g. CuSO<sub>4</sub> cryst. in 1 l. H<sub>2</sub>O and 20 g. SnCl<sub>2</sub> in 1 l. To standardize, b. 10 cc. of the Cu soln. with 25 cc. HCl and add SnCl<sub>2</sub> until it is decolorized, then add 3 cc. of a soln. of KNO<sub>3</sub> containing 0.01 N<sub>2</sub>O<sub>4</sub>. Titrate with the SnCl<sub>2</sub> soln. until decolorization ensues. In the analysis, decolorize before absorption, and titrate to decolorization after, in the same manner. 1 g. SnCl<sub>2</sub> = 0.484 NO<sub>3</sub>. NO<sub>3</sub> × 0.30449 = N. E. WALLER.

New Method for Determining Halogens in Organic Compounds. T. St. Warunis. Athens. Chem. Ztg., 35, 906-7.—After giving an extended bibliography of other methods, W. describes his method as follows: Mix 0.203 g. of the powdered substance with 10 g. KOH + 5 g. Na<sub>2</sub>O<sub>2</sub> in a large Ni crucible. Cover and heat in an air bath at 75-80° to incipient fusion; then heat over a small flame till completely fused, and maintain in fusion for some time. Take up in H<sub>2</sub>O, acidify with HNO<sub>2</sub>, filter if necessary and ppt. or titrate with AgNO<sub>2</sub>. If I is present, add a few drops of H<sub>2</sub>SO<sub>2</sub> to the soln. of the fusion. No KCN was formed in the treatment of nitrogenous compds. No analytical data are given.

New Modification of the Forensic Chemical Detection of Blood. O. VON FURTH. Z. angew. Chem., 24, 1625-8.—This test is a combination of Leers (monograph) and the leuco-malachite-green reagent (C. A., 5, 2477). The blood-stained object is digested with 50% KOH and EtOH. The aq. ext. is shaken out with pyridine and a piece of filter paper moistened with this pyridine ext. is tested in the usual way with the leuco-malachite-green reagent.

L. J. ROGERS.

Delicate Reaction for Formic Acid. G. DENIGES. Bull. soc. pharm. Bordeaux, 51, 151-4; Pharm. J., 86, 773.—To 5 cc. of dil. formic acid (1 or 2 drops to 5 cc. H<sub>2</sub>O) add as many drops of a 1: 5000 soln, of methylene blue as cc. of the formic acid soln. taken (use 2 or 3 times this amt. for acid cones. above 1%). Boil this soln. and add immediately as many drops of a 36-40° Bé. soln of NaHSO<sub>2</sub> as cc. of formic acid used. (5 drops in this case). On shaking, the blue color disappears more or less rapidly, according to the amt. of acid present, 1 mg. per cc. being thus easily detected. To further increase the delicacy of the test, add 5 drops of a 1: 10,000 soln. of methylene blue to 5 cc. of the liquid under exam. and to 5 cc. of H<sub>2</sub>O in test tubes. Immerse the tubes in b. H<sub>2</sub>O 2 or 3 min. and add to each simultaneously 5 drops of the NaHSO<sub>2</sub> soln., immerse again in b. H<sub>2</sub>O for not longer than 20-30 sec. and exam. If the decolorization is the same in both tubes less than 0.20 g. per l. formic acid is present. If formic acid be present, the tube containing it will be completely decolorized. The formic acid soln. should be acid. By distilling with H<sub>2</sub>PO<sub>4</sub> and evapg., formic acid may be detected in a liquid containing only a few mgs. per l. D. shows that the decolorization is due to the powerful reducing action of the H.S.O. formed from the NaHSO, by the formic acid. V. K. CHESNUT.

Preparation of Neutral Ammonium Citrate Solutions by the Conductivity Method. R. A. Hall. Univ. of N. Carolina. J. Ind. Eng. Chem., 3, 559-63.—Exactly neutral solns. can be readily prepared by this method and then diluted to the required d.

A. R. MIDDLETON.

Determination of Alkali in Arsenical Dip-fluids. L. COHEN. Chem. Lab. Dept. Agr., N. S. W. J. Proc. Roy. Soc., N. S. W., 64, 77-9.—The direct titration of dip-fluid being rather complicated by the presence of foreign matter, the following method was submitted by C. as being both rapid and accurate. To 50 cc. dip-fluid run in

5 cc. N H<sub>2</sub>SO<sub>4</sub>, stir, filter through a dry filter, transfer 10 cc. of the filtrate to a beaker and dil. with 100 cc. H<sub>2</sub>O. The soln. is now colorless. Titrate with 0.1 N NaOH using methyl orange for indicator. If p is no. g. Na<sub>2</sub>CO<sub>3</sub> per 100 cc. of dip-fluid and n is no. cc. 0.1 N NaOH used, then p is equal to 0.053  $\times$  (10 — 11n/10).

A. V. DE LAPORTE.

Determination of Nitrogen in Commercial Ammoniates of High Nitrogen Content. P. RUDNICK, C. H. JONES, F. C. ATKINSON, AND F. L. PARKER, JR. J. Ind. Eng. Chem., 3, 691-9.—This is the first report of the committee on N, Div. of Fertilizer Chemists, Am. Chem. Soc., and is a summary of their work and the conclusions drawn from the numerous results reported by 48 cooperating labs.

A. V. DE LAPORTE.

Determining Iron Oxide in Cement (GOLUBINZEFF). 20.

Analysis of Lime-nitrogen (KAPPEN). 15.

Determination of Volatil Aliphatic Acids (EDELSTEIN, WELDE). 11.

Microchemical Determination of Iron (Brown). 11.

Estimation of Iron Oxide in Cement (GOLUBINZEFF). 20.

Potassium Ferrocyanide (SCHRÖDER). 6.

Precipitation of Lead Carbonate (HERZ). 6.

Elaidin Reaction (FOKIN). 27.

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# 8. MINERALOGICAL AND GEOLOGICAL CHEMISTRY.

#### ROGER C. WELLS.

New Minerals. A. Schwantke. Fortsch. Min. Krist. Petrog., 1, 159-80.—A list up to 1910 with brief descriptions.

R. C. W.

Crystallization and Solution Velocities. A. RITZEL. Jena. Forisch. Min. Krist. Petrog., 1, 86-98. R. C. W.

The Phase Rule and its Application to Mineralogical Questions. R. MARC. Jena. Fortsch. Min. Krist. Petrog., 1, 99-128. R. C. W.

The Causes of Coloration of Tinted Minerals and the Influence of Radium Rays upon the Color. R. Brauns. Fortsch. Min. Krist. Petrog., 1, 129-40; cf. C. A., 5, 448. R. C. W.

Muthmannite, a New Mineral. FERRUCCIO ZAMBONINI. Palmero. Z. Kryst.

Mis., 49, 246-9.—Muthmannite resembles krennerite very closely in its crystal habit and paragenetic association. The crystals are tabular and more or less elongated in one direction. It shows a perfect cleavage parallel to the long direction. Its habit, therefore, differs from that of krennerite which is prismatic and elongated in the direction of the C axis and has a perfect basal cleavage. The color of the freshly fractured mineral is grayish white but the tarnished surface is brass-yellow. Hardness, 2; powder, iron gray. In an ign. tube it decrepitates but slightly, behaving like sylvanite. It is largely sol. in HNO<sub>2</sub>, leaving a considerable quantity of Au. When treated with HCl it gives a larger ppt. of AgCl than does krennerite. The following analysis was made by C. Gastladi: Au 22.90, Ag 26.36, Pb 2.58, Te 46.44, total 98.28. After removing AgCl, Au and Te were pptd. by oxalic acid and SO<sub>2</sub>, resp. The small quantity of Fe and Cu were not detd. and account for the low total. The formula is (Ag, Au)Te. The expts. of Pellini and Quercigh show that krennerite will not form a soln. poor in Ag while muthmannite will form if sufficient Ag is present.

E. S. Larsen.

Apophyllite from Tergarhorn, Iceland. O. B. Böggild. Copenhagen. Z. Kryst. Min., 49, 239-45.—The apophyllite crystals from Tergarhorn, Iceland, are 1.5-3 cm. long and show the plane faces (001), (113), (111), and (100), and other curved and striated faces. The angle (001): (111) averaged 60°  $22\frac{1}{2}$ ; therefore c = 1.2435. The apophyllite is deposited on delessite and on it are deposited crystals of desmine. Some calcite and laumontite crystals are present. Each crystal of apophyllite is made up of two distinct parts. Generally the corrosion is irregular, but in some cases the embayment starts from the center of the face (100) and passes through the whole crystal. Some calcite crystals occur in the embayments. The series of processes involved in the formation of the mineral appears to have been as follows: (1) Deposition of the delessite, which continued in slight amt. to the end; (2) deposition of the central part of the apophyllite crystals; the faces formed were (100), (130), (120), (111), and probably (001); (3), corrosion of the apophyllite crystals; (4) deposition of the calcite crystals in the pits of the apophyllite; (5) deposition of the small ends of the apophyllite crystals; (6) deposition of the desmine.

E. S. Larsen.

Determination of the Crystallographic Constants of some Artificial Apatites. A. DE SCHULTEN. Compt. rend., 152, 1404-6.—The minerals were prepared by fusing chlorides or bromides with the tribasic phosphates or arsenates, or with the neutral phosphate of  $NH_4$ . The magnitudes of certain angles and of the axis c in the artificial apatites of Ca, Sr, Ba and Cd are given, and compared with those of the natural apatite as well as those of pyromorphite, mimetite, and vanadinite. It is found that the axis C diminishes in length as the at. wt. of the metal decreases, as the at. wt. of the halogen increases, and also when As is substituted for P.

Edw. Wolfsensky.

Crystallographic Investigation of Certain Iodosulfides. Demaisseux. Bull. soc. franc. min., 32, 287-96.—D. gives the results of investigations into the physical and optical properties, and % comp. of CHI<sub>2.3</sub>S<sub>2</sub>, SbI<sub>2.3</sub>S<sub>2</sub>, AsI<sub>2.3</sub>S<sub>3</sub>, C<sub>2</sub>I<sub>4.4</sub>S<sub>3</sub> and SnI<sub>4.2</sub>S<sub>3</sub>.

C. G. GILBERT.

Preliminary Crystallographic Observations on Tetrahydrated Cerium Selenate. ARISTIDE ROSATI. Ist. min. r. univ. Roma. Atti accad. Lincsi, 20, II, 149–50.—Ce<sub>3</sub>(SeO<sub>4</sub>)<sub>2</sub>.4H<sub>2</sub>O (Cingolani, C. A., 2, 2568) forms trimetric, hexagonal prisms, (001), (010), (110), and occasionally (001), (110); a:b=0.5834:1. The faces are strongly striated and corroded. Chas. A. ROUILLER.

The Determination of the Principal Birefringence from a Section Showing the Optic Axes. HERMANN TERRECH. Min. petrog. Mitt., 29, 520-2.—The author de-

rives the formulas:  $\alpha = \pm \beta \sqrt{\beta}/2r \sin^3 \gamma + \beta'$  and  $\gamma = \pm \beta \sqrt{\beta}/2r \cos^3 \gamma + \beta$ , where  $r = l \ K/d \sin \varphi \sin \varphi'$ , l = the displacement of the band in the Babinet compensator; K = the constant of the Babinet compensator; d = the thickness of the section;  $\varphi$  and  $\varphi'$  are the angles between the normal to the section and the 2 optic axes. The empirical formula of Biot is a satisfactory approx. for minerals with a low birefringence,  $(1/\alpha_1^2 - 1/\gamma_1^2) = (1/\alpha_1^2 - 1/\gamma_1^2) \sin \varphi \sin \varphi'$ . E. S. LARSEN.

Law of Mineral Association form the Standpoint of the Phase Rule. V. M. GOLD-SCHMIDT. Z. anorg. Chem., 71, 313-22.—For the law of mineral formation the phase rule must be used, not in its most general form, but in a specialized form, which may be stated thus: The maximal number, n, of solid minerals which can coexist in stable equil. is equal to the number, n, of the single components contained in the minerals. This holds for any arbitrary pressure and temp., whereas when one of these conditions is fixed, as at a transition point, n + 1 minerals can coexist. A number of cases is discussed, illustrating the application of this mineralogical phase rule.

EDW. WOLESENSKY.

Association of Rutile and Cyanite from a New Locality. Thomas L. Watson and Joel, H. Watkins. Am. J. Sci., 32, 195-201.—Rutile occurs in Charlotte County, Virginia, as inclusions in the form of fine grains (often microscopic), principally in a cyanite schist, but also in a thinly foliated quartz-sericite schist. It is associated with and inclosed in the substance of both quartz and cyanite, but is more abundant in the latter. Its relation to the quartz and cyanite precludes its formation subsequent to that of these minerals. The cyanite is partly altered to muscovite. E. W.

A New Occurrence of Pearceite. F. R. VAN HORN AND C. W. COOK. Am. J. Sci., 31, 518-24.—This mineral has been found in the Veta Rica mine at Sierra Mojada, state of Coahuila, Mex. About 200 lbs. of crystals, associated with such minerals as native Ag, argentite, proustite, and erythrite, were taken from a fault plane. An analysis calc. to rational basis closely approximates the formula (AgCu)<sub>16</sub>As<sub>2</sub>S<sub>11</sub> instead of the generally accepted one (AgCu)<sub>16</sub>As<sub>2</sub>S<sub>12</sub>. The physical properties, crystal form and pyrognostics of the mineral are fully described.

R. J. WYSOR.

A Discussion of the Formulas of Pearceite and Polybasite. F. R. VAN HORN. Am. J. Sci., 32, 40-4.—Only 5 analyses of pearceite have been published prior to the one given in the preceding abstr. A critical study of these results corroborates the formula (AgCu)<sub>18</sub>As<sub>2</sub>S<sub>13</sub> instead of (AgCu)<sub>18</sub>As<sub>2</sub>S<sub>12</sub> formerly proposed. A similar review of analyses of polybasite confirms the observance that it is isomorphous with pearceite and is represented by the formula (AgCu)<sub>16</sub>(AsSb)<sub>2</sub>S<sub>11</sub>. R. J. WYSOR.

A New Synthesis and New Occurrences of Covellite. A. F. Rogers. School of Mines Quart., 32, 298–304.—Covellite was prepared by metathesis between sphalerite and an aq. soln. of CuSO<sub>4</sub> in a hard glass tube in an atm. of CO<sub>3</sub> at 150–60°. The principal sources of covellite are named.

R. J. Wysor.

The Reproduction of Natural Gem Stones. L. C. WHITON, JR. AND L. C. WHITON. Mining Sci., 64, 60-1.—Various methods for the production of artificial diamonds and Al<sub>2</sub>O<sub>2</sub> gems, including the application of Ra, are reviewed. R. J. WYSOR.

Some Notes on Pyrite and Marcasite. E. B. Wilson. Min. Sci., 64, 156-7.—
The unreliability of much of the literature dealing with these minerals is emphasized.

Information known to be accurate relating to their distinguishing features is summarized.

R. J. Wysor.

The Electrical Conductivity and Behavior of Diamonds at High Temperatures. C. DOELTER. Monatsh., 32, 275-98.—Cond. tables are given showing resistances at various temps. between 880° and 1290° in an atmos. of H and of O, although results in the latter case proved unsatisfactory because of corrosive action taking place

at higher temps. The curve plotted to temp. and resistance shows a marked diminution in resistance to 1200°, a more gradual diminution thence to 1240°, followed by a rather strongly increasing resistance to 1260° when the falling off begins anew. It was possible to heat the diamond to 2000–2500° without distinguishable effect, and D. regards it as questionable whether temp. alone will suffice to effect alteration to graphite.

C. G. GILBERT.

A Notable Brazilian Diamond. ORVILLE A. DERBY. Am. J. Sci., 32, 191-4.—A description of the form and dimensions of a large diamond found in the Bagag =m river diamond district, in the headwater region of the Paraba River.

Edw. Wolesensky.

Bologna Stone. III. L. VANINO AND E. ZUMBUSCH. J. prakt. Chem., 84, 305–17.—In good phosphorescent stones, the % of S can vary from 12 to 33%. The presence of polysulfides favors the phosphorescence, the pure monosulfide stone showing weak phenomena. The storing of the product in atmospheres of diff. gases and the mixing with organic dyes did not increase the light effect.

A. A. KLEIN.

The Chemical Composition and Crystallization of Parisite and a New Occurrence of it in the Granit Pegmatites of Quincy, Mass. Chas. Palache and Ch. H. Warren. Am. J. Sci., 31, 535-57.—The various discoveries of parisite and the meager data relating to its crystallography are reviewed. In the Quincy area it is found in aegeritebearing rocks and from other observed occurrences it appears certain that parisite or synchisite and cordylite are characteristic pneumatolitic minerals of the riebeckite aegerite rocks. The pegmatite pipes in the surrounding granit carry practically all of the parisite. The crystals, particularly plentiful in cavities, average 1-3 mm. in length, the max. being about 2 cm. A minute description of the crystallography and optical properties of the mineral is given. Corroborated analyses of the crystals show the following comp.: CO<sub>2</sub> 24.16%, F 6.56, Ce<sub>2</sub>O<sub>3</sub> 30.94, (LaDi)<sub>2</sub>O<sub>3</sub> 27.31, Fe<sub>2</sub>O<sub>3</sub> 0.32, CaO 11.40, Na<sub>2</sub>O -0.30, K<sub>2</sub>O -0.20, gang 1.02. The authors believe that synchisite and parisite are the same mineral. Notes also appear on the occurrence of microcline, riebeckite, aegerite, ilmenite, octahedrite, fluorite and wulfenite in the sand area. R. J. Wysor.

The Pegmatites of the Riebeckite Aegerite Granit of Quincy, Mass., their Structure, Minerals and Origin. CH. H. WARREN AND CH. PALACHE. Proc. Am. Acad., 47, 125-68; cf. C. A., 4, 159.—The igneous rocks of this area are intrusive in Cambrian strata. They consist essentially of granit, granit porphry, quartz-alkaline feldspar and rhyolite. Elsewhere narrow bands of pegmatite have been discovered. The most important occurrences of this pegmatite in the granit quarries are in pipe-like form. The first is nearly vertical, roughly cylindrical and consists of 3 concentric radial shells. The outer 2-5 in. in thickness is differentiated from the surrounding granit by a greater segregation of its constituents and by a greater abundance (also with a different ratio) of aggerite and riebeckite. The next zone, 1-2 in. wide, is fine graphic-granite, containing a few prisms of aegerite and riebeckite. The main interior portion consists essentially of a pegmatite or coarse-grained aegerite granit, with some riebeckite and many other accessory minerals. Minute mineralogical data as to the 3 zones is given. One of the other pegmatite pipes reveals 4 zones. The outer 2-5 in. thick, is finer in grain and richer in dark silicates than the surrounding granit. The next formation, 2-4 in. thick, consists substantially of microcline albite microperlbite (60%) and quartz 30%. The third zone is a close-grained pegmatite containing many accessory minerals. There is a large central mass of quartz. A discussion of chem. comp., sequence of crystn. and origin of the pegmatite is followed by detailed notes on the microscopic properties and general physical characteristics of the individual minerals. Several microphotographs of representative mineral groups in the pipes appear.

R. J. Wysor.

Baddeleyite and Pyrrhite from Monte Somma. Preliminary Communication. Ferruccio Zambonini. Ist. min. r. univ. Palermo. Atti accad. Lincei, 20, II, 129-30. —The minerals were found in a block of sanidinite particularly rich in Zr. The baddeleyite appears in isolated or clustered crystals, elongated along c and tabular along (100). The forms a (100), c (001), m (110) and q(011) are always present, r (101) rarely. It shows cleavage along (001), (010) and (110). The pyrrhite occurs in isotropic, octahedral crystals, reddish brown by reflected, dark orange-yellow by transmitted light.

Chas. A. Rouiller.

New Forms of Elba (Beryl). FEDERICO MILLOSEVICH. Atti accad. Lincei, 20, II, 138-44.—The following new forms were discovered in the collection of Elba minerals in the Florence Museum: Bipyramidal of the 1st order; (1013), new for the species; (1.0.1.14), new for Elba. Bipyramidal of the 2nd order: (3.5.10.7), new for Elba; (3365), (4489), (1.1.2.10), new for the species. Dodecagonal bipyramidal: (3255), (5499), (8191), all new for the species. C. A. ROUILLER.

Chemical Composition of a Zinc Blende. II. RAMON LLORD Y GAMBOA. Anales soc. espa#. fis. quim., 8, 413-21.—This blende from Picos de Europa is a mixed solid soln. with an isomorphous mixt. and a mixt. not isomorphous irregularly distributed in the cryst. mass; this unequal distribution can be readily observed in the case of the chromogenic substance (which is very probably of a ferroso-ferric nature). In cryst. masses of some size, besides Zn and Cd, there are always found Fe, Cl, Ca, Na, and organic matter, while Mn, Pb, Al, K, and Li are variable. Li, K, Na, and Ca (present as chlorides) may be so completely washed out by hot H<sub>2</sub>O from the powdered blende as to leave no trace detectable by the spectroscope. This same washed powder (from which chlorides and H<sub>2</sub>O sol. organic matter have also been removed) exhibits between 300° and 400° the irreversible phenomenon of thermoluminescence. On evapg. the filtrate from the washed blende and igniting, a resinous odor was observed, a loss in wt. corresponding in 1 case to 0.007% organic matter.

H. S. Paine.

Microlitic Rocks of the Boucle du Niger. Henry Hubert. Compt. rend., 152, 1606-8.—In the Boucle du Niger, besides the gneisses of Dahomey and the schists and dibases of French Guinea, there occur also microlitic rocks. Classed as trachytes are compact rocks without quartz and rich in K, characterized by phenochrysts of plagioclase in a matrix rich in microlites of orthoclase. The dacites are very light gray rocks with phenochrysts of corroded quartz, oligoclase-albite, and less often hornblende or mica. The andesites are very abundant and nearly all are characterized by phenochrysts of plagioclase and metasilicates, while the matrix is invaded by secondary minerals as quartz, calcite, epidote, etc. The augite is usually intact, while other minerals are altered. Ba salts are rare and much altered, and can be recognized only by the original minerals (plagioclase, pyroxenes) which here and there have been preserved. Tuffs and breccias are also present, as well as amphibole schists resulting from the crushing of diabases. These types are quite extensive. E. Wolfsensky.

Materials of the Explosive Rhyolitic and Trachytic Eruptions of Mont-Dore. A. MICHAEL LEVY AND A. LACROIX. Compt. rend., 152, 1200-4.—The greater part of Mont-Dore consists of projected material which has been shown to be divided into two series, a lower and an upper cinerite. The upper cinerite, by the character and comp. of its material, gives evidence of at least two separate phases of eruptive activity, the material of the first being of a rhyolitic, that of the second of a trachytic nature Detailed descriptions as well as chem. analyses are given.

EDW. WOLESENSKY.

Graphite as a Primary Rock Constituent. A. N. WINCHELL. Mining Sci., 64,

148.—Graphite has probably originated in nature in several ways. In certain rocks as pegmatites, it must be recognized as a primary constituent; hence the C must have existed in some form in silicate solns. Neither hydrocarbons nor metallic carbides are probable sources of graphite. The theory is advanced that C has been deposited during cooling below 1000° by the reaction of H with CO<sub>2</sub> or CO. All of these gases are known to exist in magmas. On further cooling below 600° the deposited C would be expected to crystallize as graphite.

R. J. Wysor.

The Nature of Volcanic Action. R. A. Daly. Proc. Am. Acad., 47, 48-122. —The following are indicative features of this article which is too elaborate and diverse for a brief abstract. A fundamental geological postulation is that the earth is exteriously composed of successive shells. Underlying the interrupted sedimentary shell is a continuous granit zone. Still deeper is an emptible basaltic mass. All igneous action is the result of mechanical intrusion of substratum basalt into the overlying shell and this process is denominated abyssal injection. The necessity of classifying the important gases at volcanic vents is emphasized. The article is chiefly concerned with central eruptions. Explanation is offered for the dormancy and related periodicity of certain vents. Application of various theories is made.

R. J. WYSOR.

Ferriferous and Carboniferous Deposits in Ogliastra (Sardinia). E. CORTESE. Genoa. Rass. min., 35, 49-51.—Description of the F and carboniferous deposits in the desert regions of Perdasdefogu, Tertenia, Jerzu, Ulassai, Osini, Ussassai, Sadali and Esterzili. The carboniferous material seems to be a lignite and that containing iron is of the swamp ore type, containing 55-7% Fe, 0.12% S, 0.80 P, 5.5% Al<sub>2</sub>O<sub>3</sub> and 1.5% SiO<sub>5</sub>.

C. A. ROUILLER.

Preliminary Notes on some Igneous Rocks of Japan. S. Kozu. J. Geol., 19, 555-60, 561-6, 566-76.—I. Petrographical-chemical description of a soda-trachyte found on Matsu-Shima and Kakara-jima, two islands N. W. of Yobuko, prov. Hizen, Kyushu. Sep. analyses were made of the rock and of the porphyritic anorthoclase. In the quant. system the rock would be classified as a laurvikose. There is a close chem. resemblance between this type and a pantelleritic trachyte. II. At Kasa-yama, near Hagi City, prov. Nagato, a quartz-basalt was encountered. The quartz occurs as a porphyritic constituent with an av. diam. of about 2 mm. Each grain of quartz is fringed with a reaction-border, consisting of elongated prisms and grains of augite imbedded in brown glass. The analysis reveals a striking similarity to a quartz-basalt described by Diller from Lassen Peak, Cal. Under the quant. system it would be classified as a bandose. III. A description with analyses of alkali-feldspar-bearing basaltic rocks from the northern part of Kyushu.

The Evolution of Limestone and Dolomite. II. EDWARD STEIDTMANN. J. Geol., 19, 392-429; cf. C. A., 5, 2796.—Dolomites develop in the sea rather than by metamorphism of limestones after their emergence, and the decline in dolomite in ascending the geological column indicates a change in the conditions of deposition. There is evidence for a change in the ratio of Ca and Mg contributed to the sea from the lands. The increase in the proportion of Ca to Mg in streams is believed to be due to selective processes, which are cumulative with time, the Ca being transported in soln. to the sea while the Mg is retained in the residuals. Erosion removes the residuals towards the margins of the land and during continental expansion a proportion is swept into the deep sea and lost. During oceanic expansion max. deposition of limestone and dolomites takes place on the submerged lands. The % of Ca pptd. is higher than that of Mg. With the progressive elimination of clastics and Mg from the lands with geological time, and in their place the gradual accumulation of Ca in the form of lime-

stone, the proportion of Ca to Mg contributed by rivers to the sea has increased with time.

W. F. Hunt.

Mineralization of the Gold-bearing Lode of Passagem, Minas Geraes, Brazil. ORVILLE A. DERBY. Am. J. Sci., 32, 185-90.—Recent investigations seem to show that the inclusions in the Au-bearing lode of the Passagem mine are of secondary origin. This is concluded from the presence of inclusions of sericitic mica, more or less heavily charged with calcite, which give evidence of an original feldspathic mineral. In the vicinity of these the quartz shows fissures filled with tourmaline, pyrites, and other minerals. Evidence is also given showing that the formation of the tourmaline and of the sulfides took place independently, and that the former preceded the latter.

EDW. WOLESENSKY.

Origin of Certain Bonanza Silver Ores of the Arid Region. C. R. KEYES. Bull. Am. Inst. Mining Eng., 55, 541-58.—The occurrence of rich AgCl gossau ores in desert tracts has not been explained satisfactorily. Weathering action in deserts must be chiefly mechanical. Secondary enrichment at low levels may not be due to leaching from the vein above but from oblique fault or joint planes. Not only AgCl but chlorides of other metals such as Cu and Fe have been found in arid regions. In some instances probably Au also has been derived from the chloride. It is believed that these metallic chlorides were formed on the surface by action of alkali chlorides from the desert. Subsequent soln. and percolation has carried them to lower levels. Gang clays frequently contain notably higher precious metal values than the adjacent ore veins. This is explained by the dialytic action of clays in selective adsorption of the metallic salts and by the more potent influence, the reduction of metallic salts in ground waters by alkali silicates.

R. J. Wysor.

The Classification of Sand Grains. A. W. Graham. Science, 33, 1005-7.—Parallel work with W. H. Sherzer reveals close agreement in the classification systems employed for the various types of sands. G., however, has added one or 2 sub-types to Sherzer's classification. An improved standard terminology is offered for naming the several types.

R. J. Wysor.

Structure of the Earth's Crust in German East Africa. E. Kohlschütter. Nachr. kgl. Ges. Wiss. Göttingen, 1911, 1-40.—The author gives an elaborate mathematical interpretation of the physical forces involved and of their effects. C. G. Gilbert.

Solubility of Clay and Flint in Feldspar (PURDY). 19.

Geochemical Interpretation of Water Analyses (PALMER). 14.

Application of the Phase Rule to Mineral Silicates (GOLDSCHMIDT). 2.

Fermann, A. and Goldschmidt, V.: Der Diamant. Heidelberg: 274 pp., 12:50 M.

KAYSER, E.: Lehrbuch der Geologie. 4 Aufl. Stuttgart: 8°, 798 pp., 20 M.

Marc, R.: Vorlesungen tiber die chemische Gelichgewichtslehre und ihre Anwendung auf die Probleme der Mineralogie, Petrographie u. Geologie. Jena: 218 pp., 5 M.

ODENDALL, L.: Die Kupferlagerstätten in Nordamerika. Bonn: 8°, 63 pp., 1.20 M.

TANNHIUSER, F.: Mineralogische Wandtafeln. No. 1, 5 M.; No. 2, 5 M.; No. 3, 8 M. Leipzig: Paul List.

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# o. METALLURGY.

### WILLIAM BRADY, WILLIAM T. HALL.

Observations on Nickel Mining in Canada and its Relation to the Manufacture of Nickel Anodes. G. L. WALLACE. Brass World 7, 349-51.—The average of the workable metals in the ore of the Creighton mine is 4-5% of Ni and 1.5-2% Cu. The roasted ore is converted into Ni mat which contains about 80% Ni and 20% Cu.

ROBERT KANN.

The Application of Graphite to the Production of Crucibles for Melting Metals. II.

A. HARNIG. Brass World, 7, 351-2.—Various practices are enumerated to protect the crucibles from the chem. action of the charge, from the constituents of the shell, to prevent the absorption of H<sub>2</sub>O, or to increase the resistance of the lining against high temps. The most recent improvement in the manuf. of graphite crucibles is the process of H. Putz, of Perssau, which consists in coating the surface of the crucible with rosin, tar, paraffin or a similar product. H<sub>2</sub>O is thus prevented from penetrating the crucible.

ROBERT KANN.

The Deadwood Mill at Mogollon, N. Mexico. J. W. Kniffin. Eng. Mining J., 92, 744-5.—The mill treats Au and Ag sulfide ores.

ROBERT KANN.

The Cupellation of Lead Bullion. R. F. COOLIGE. Eng. Mining J., 92, 756-7.

—Details of expts. for the recovery of Au from the by-products of Zn ppt., refining by smelting with litharge and cupelling the bullion.

ROBERT KANN.

First Aid to Mining Machinery. I. H. M. Lane. Eng. Mining J., 92, 758-60.—The method of making green sand molds is described. II. Ibid., 804-5.—A description of the construction and operation of a small cupola for melting Fe for casting; notes on the selection of m. mixts. and influence of various constituents upon the quality of Fe.

ROBERT KANN.

General Rules for Annealing Rolled and Forged Carbon Steel. H. M. Howe, et al. Eng. News, 66, 188-9.—Rules drawn by the Committee A-4 of the Am. Soc. Testing Materials on "Heat Treatment of Iron and Steel." 1. Purpose: Annealing removes coarseness of grain and serious internal stresses due to rolling or working the metal. (2) Method of Heating: The temp. should be held long enough at the annealing point to insure complete heating. (3) Control of Temps.: Use of trustworthy pyrometers. (4) Control without the Use of a Pyrometer: Strong lights surrounding the furnace should be avoided. Allowance must be made for brighter surroundings by day than by night. (5) Magnetic Indications: With steel of 0.50-0.90% C, the metal suddenly ceases to be magnetic at the annealing temp. (6) Annealing Temp.: In general, the higher the C the lower the annealing temp. The following ranges are given: C less than 0.12%, 875-925°; C, 0.12-0.29%, 840-870°; C, 0.30-0.49%, 815-840°; C, 0.50-100.0%, 790-815°. (7) Care in Heating: The flames should never touch any part of the object heated. (8) Cooling: The higher the C, the slower must be the cooling. The slower the cooling, the softer and more ductil the metal will be, and the lower its tensil strength, elastic limit and yield point. (12) To give an unusually high ductility with tensil strength and elastic limit, quench in water or oil. Anneal within a few hours after quenching, maintaining the temp. at least 100° during the interval. For high elastic limit and tensil strength, intermediate elastic limit and tensil strength, and for greatest ductility, anneal at 500°, 600°, and 725-750°, resp. L. A. Touzalin.

The Process of Zinc Distillation. Geo. A. Wettengel. Met. Chem. Eng., 9, 198-201; see C. A., 5, 1730.

L. A. T.

The Counter-current of Continuous Agitation, Decantation, and Dilution Applied

to the Cyanide Process. John E. Rothwell. Met. Chem. Eng., 9, 439-41.—The present agitation method of cyanidation requires the mixt. of a barren cyanide soln. with the ore at max. value. By continuous agitation, dissoln. is accomplished and the metal transferred to the soln. The rate of dissoln. proceeds more slowly as chem. equil. is approached. A re-treatment of the charge after removal of the first soln. is sometimes necessary. The new method, described in detail (graphic flow sheet), is the reverse of the above, i. e., the solvent lowest in metal value and highest in free cyanide is applied to the ore after the greater value in metal has been dissolved. This soln. is then sepd. and applied to ore of higher value, and so back up the line of treatment until the highest grade of soln. is in contact with the ore at max. value. It is then sepd., the metal content is pptd. and the barren soln. used again. The system may be applied to the treatment of ores other than Au and Ag where a hydrometal-lurgical method can be applied.

L. A. Touzalin.

Notes on the Treatment of Mill Concentrate. R. LINDSAY. J. Chem Met. Min. S. Africa, 12, 2-6.—A description of the methods used at the Geldenhuis Deep reduction works. The details can hardly be understood without the accompanying diagram. The scrape is ground in barrels for 4 or 5 hrs., cleaned over a stationary Cu plate, and then run into a tank having a baffle plate 18 in. from the inflow end, the overflow being conducted to a settling tank. This arrangement acts as a trap for Hg and amalgam. From that the material runs through a riffled launder to the settling tank above mentioned, which receives the overflow. The overflow from this last runs into another larger settling tank, before it settles clear enough to be run to waste. The slimes from these tanks are cyanided in a cone-shaped tank by the Crosse method (cf. C. A., 4, 739). The charge is agitated with air 12 hrs. before beginning to wash with 0.02 per cent. KCN soln. Washing is continued at the rate of half a ton of soln. per hr. 1800 tons of concentrate requires 6 tons of soln. After a total treatment of 24 hrs. the residue assays 4 dwt. and the soln. 0.08 dwt. per ton. The consumption is 2.75 lbs. of 120% KCN, as the material is very refractory. The air for agitation is obtained through a branch pipe from an Ingersoll-Rand compressor. The consumption is 7 cu. ft. of free air per min. at a cost of 5 d. per ton of concentrate. Cost of tube milling 15 s. Of cyaniding 11 s. Total cost per ton of concentrate 26 s.

E. WALLER.

Experiments in the Treatment of Accumulated Ore Slime by Air Lift Agitation. J. E. R. ADENDORFF. J. Chem. Met. Min. S. Africa, 12, 6-13.—The cyanides in accumulated slime from blanket ore appear to be free H<sub>2</sub>SO<sub>4</sub> and partially oxidized Fe-S compds. Experiments with bottle shaking tests, and in a small Pachuca tank capable of treating about 100 lbs. of slime at a time indicated that success depends on maintaining a high alkalinity during the first 3 hrs. of aeration, and that CaO should be added to the slime before treating with the KCN and that (AcO)<sub>2</sub>Pb improves the extraction. The minimum alkalinity allowed should be about 0.012% CaO. More than half the Au was dissolved out during the first hr. In a 30-hr. run no advantage was gained by prolonging the treatment after the 18th hr, and the alkali and free HCN strengths remained unaltered. In this run the original slime contained 2.33 dwt., the tailings 0.20 dwt.

Suggestions on Cyanide Practice. J. B. STEWART. Mining Sci. Press, 103, 254.—The author suggests sep. treatment of natural slimes, as well as their sepn. before final regrinding. Stage grinding is also recommended. EDW. WOLESENSKY.

Specifications for Spelter. Anon. Mining Sci. Press, 103, 289.—Specifications for spelter as adopted by the Amer. Soc. for Testing Materials. E. WOLESENSKY.

Combination Melting and Cupelling Furnace. WILL. H. GOGHILL. Mining

Sci. Press, 102, 819.—The furnace described is of the ordinary tilting type, fired with either gas or gasoline, with burner fastened to the furnace so that the latter may be tilted without interrupting the blast. In this way the furnace may be used for cupelling as well as for melting. Temps. as high as 1900° F. may be attained. E. W.

History of the Cyanide Process. J. McCombin. Mining Sci. Press, 102, 850. Edw. Wolfsensky.

The Ternary System: Silver-Zinc-Lead. A Contribution to the Theory of the Parkes Process. R. Kremann and F. Hofmeier. Monatsh. Chem., 32, 563-96. -This system of alloys really comprizes 5 distinct ternary systems, Pb-Zn-Ag-Zn. Pb-Ag<sub>2</sub>Zn<sub>4</sub>-Ag<sub>2</sub>Zn<sub>4</sub>, Pb-Ag<sub>2</sub>Zn<sub>4</sub>-AgZn, Pb-AgZn-Ag<sub>2</sub>Zn<sub>2</sub>, and Pb-Ag<sub>2</sub>Zn<sub>2</sub>-Ag. The authors have studied each of these systems as well as all the binary systems involved that have not been investigated previously. In the Parkes process Pb-Zn-Ag.Zn. only is concerned. The Zn is kept between 0.8 and 1.5% so that as the melt cools the primary deposition consists of mixed crystals of Zn and Ag, Zn<sub>a</sub>. As these are deposited, the eutectic curve between crystals rich in Zn and Pb is approached and when this is reached, pure Pb deposits. The deposited crystals are removed, more Za is added to the melt and the comp, of the remaining alloy gradually approaches the binary system of Pb and Zn and the Ag content is reduced under favorable conditions to 0.005%. In working up the crystals containing Ag, they are heated to a higher temp. First of all, the ternary eutectic melts; it consists of Pb and mixed crystals of Zn-Ag<sub>2</sub>Zn<sub>2</sub>. Finally all the Pb is melted and only crystals containing Ag and Zn remain. W. T. HALL.

The Electromotive Behavior of Ternary Alloys. (The Ternary System: Zinc-Silver-Lead.) R. Kremann and F. Hofmeier. Monatsh., 32, 597-608.—If an alloy of 2 or more metals is dipped into a soln. of an electrolyte a p. d. results between the electrode and the solution and there are 3 cases to distinguish: (1) If the alloy is a mere mechanical mixt. then the p. d. is that of the baser metal. Thus when Zn-Pb or Ag-Pb is dipped in a soln. of ZnSO, the potential difference in the former case is that of Zn toward ZnSO<sub>4</sub> soln. and in the latter case of Pb. (2) If the metals form solid solns. then the potential is intermediate between the values obtained with the individual metals and the p. d. is here detd. by the loss in free energy in the formation of the solid soln. (3) When a chem. compd. is formed the p. d. is usually, but not necessarily, between that of the individual metals. Cases 2 and 3 are met with in Ag-Zn alloys because 4 compds., Ag.Zn, Ag.Zn, Ag.Zn, and Ag.Zn, are formed which, are partially miscible with one another in all proportions and partly not. These potentials in the case of ternary Pb-Zn-Ag alloys are tabulated. The potentials obtained, in spite if varying additions of Pb, correspond to those in the binary Ag-Zn alloys. This, like the results obtained by thermic analysis, shows that Pb does not form any ternary mixed crystals with Ag and Zn. W. T. HALL.

The Equilibrium Diagram of Iron-Carbon Alloys. Of ORUFF. Metallurgie, 8, 456-64, 497-508.—In the diagram the curve OS represents the transformation of  $\gamma$ -Fe into  $\beta$ - and  $\alpha$ -Fe according to Goerens and Meyer, the curve S<sub>1</sub>E<sub>1</sub> the solubility of carbide in  $\gamma$ -Fe according to Heyn and Gutowsky, the curve SE the solubility of C in  $\gamma$ -Fe according to Ruer and Iljin, the curve EA that of C in  $\gamma$ -Fe according to Gutowsky, the curve AB that of  $\gamma$ -Fe mixed crystals in liquid Fe according to Gutowsky and to the expts. of Spieker published by Wuest. The line EBC represents the boundary for the solidus-liquidus (graphite and mixed crystals) in the stable system according to the results of Gutowsky for white and gray irons. The curve BDHI, from 1135° to 2620°, unites the solubilities detd. in the present research. The curve IY is drawn as the most probable continuation of HI in order to reach the line XZ which seps. the liquid and gaseous phases. The latter line is based upon a few rough expts.

and serves to show about where the liquid phase ends. The line E<sub>1</sub>B<sub>1</sub>C<sub>1</sub> seps. the solidus and liquidus (carbide-mixed crystals) in the metastable system and is drawn 10° below EBC. R. concludes from his expts. that besides cementite, Fe<sub>2</sub>C, which he prefers to call *triferrocarbide*, there exists a diferrocarbide, Fe<sub>2</sub>C. The equil. conc. of the triferrocarbide is always less than the satn. conc. Above 700° the carbides are endothermic compds. and at 2220° the diferrocarbide is quite strongly dissociated

into Fe and C. As the temp. is lowered, the diferrocarbide breaks down 2700 into Fe<sub>2</sub>C and C and eventually the Fe<sub>2</sub>C into 2500 Fe and C. In the metastable system, the solidi- 2300 fication takes place in 4 periods: (1) The time up 2/00 to that point when the melt reaches the equil. 1900 conc.; (2) the time during which the melt remains between the equil. conc. and the satd. conc.; (3) the time during which the carbide eutectic is solidifying; (4) the time during 1100 which the solid system is cooling. The amt. of solid 900 0 carbide formed from the liquid phase is detd. 700 3 2 chiefly by the decomp.

velocity of the carbide and the difference between the equil. conc. and the satn. conc. and is also affected by the velocity of solidification. In the solid phase the decomp. velocity of the carbide is always less than in the liquid phase. Thus, when the carbide is once formed as solids, it remains more stable in proportion as the temp. is low at which it is first deposited. Moreover, the more finely divided its condition, the more likely the carbide is to remain in metastable equil. with the surrounding solid soln. The reaction velocity of the carbide decomp. at 1135° is such that about 50% of the carbide will decompose in 2 secs. R. has developed approximation formulas for the results to be expected in the solidification of Fe-C alloys during the second and third periods.

W. T. Hall.

Metallographic and Photochemical Investigation of the System, Sulfur-Tellurium. Masumi Chikashigs. Chem. Inst., Kyoto. Z. anorg. Chem., 72, 109-18.—By thermic analysis and photochem. studies it is shown that S and Te can form no compds. with one another and that the ppts. formed by the action of  $H_2S$  upon  $H_2TeO_3$  consist of a mixt. of S and Te. In fact 2 series of mixed crystals are formed and a eutectic with 7% Te which m. at 109°. The satd. mixed crystals on the Te side contain 2% S and experience no transformation below the m. p. Fused  $\gamma$ -S dissolves about 20% Te, fused  $\beta$ -S not more than 10% Te, and solid  $\beta$ -S only 2% Te. Low temps. or light rays transform the  $\beta$  mixed crystals into  $\alpha$ -crystals and in the latter about 5% Te will dissolve. The crystals with 0.5 to 2% Te are sensitive to light rays and the greatest effect is obtained when  $\lambda = 450$ . Mixed crystals with less than 0.5% Te are scarcely influenced by light and to this class of crystals belongs the Japanese red S. W. T. H.

The Internal Structure of Martensite and Pearlite. M. Oknov. Polytechnischen Instituts, St. Petersburg. Metallurgie, 8, 539-41.—By examining the etched surfaces of specimens of martensitic and pearlitic steels after removing several successive layers about 0.001 mm. thick, the author was able to trace the form of these constituents. Martensite was found to consist of lamellar crystals instead of needles. The pearlitic structure was found to be due to bent plates of cementite embedded in ferrite.

CARLE R.: HAYWARD.

Ternary System: Silver-Tin-Lead. N. PARRAVANO. Ist. chim. r. univ. Roma. Gazz. chim. ital., 41, I, 813-32; cf. C. A., 5, 2389.—The present paper contains detailed experimental data, diagrams and photomicrographs.

CHAS. A. ROUILLER.

Conductivity of Light Aluminium Alloys (WILSON). 4.

DICHMANN, C.: The Basic Open-hearth Steel Process. London: Constable. 8°, 346 pp., 10 s., 6 d.

GRIGER, C.: Handbuch der Bisen-und Stahlgiesserei. Berlin: J. Springer. 8°, 472 pp., 20 M.

JUPTNER, J.: Das Bisnehüttenwesen. Leipzig: Akademische Verlagsgesellschaft.  $7~\mathrm{M}.$ 

KOSSMANN, W.: Ueber die wirtschaftliche Entwicklung der Aluminiumindustrie. Frankfurt a/M.: J. Baer & Co. 2 M.

Lodge, R. W.: Notes on Assaying and Metallurgical Laboratory Experiments. 3rd Ed. New York: John Wiley & Sons. 317 pp., \$3.00.

NAGEL, O.: Nagel's Gold Book. Contains a Description of the Recovery of Gold from Sea Water. New York: O. Nagel. \$2.00.

- U. S., 1,001,057, Aug. 22. R. LUCKENBACH, Colwyn, Pa. Concentrating amalgamator.
- U. S., 1,001,279, Aug. 22. E. C. KAYSER, Cincinnati, Ohio. Assignor to Procter and Gamble Co., same place. Making finely divided catalytic nickel, diffused through or deposited upon refractory material, non-pyrophoric and stable in air, by submitting inert material (or oxide or hydroxide of Ni) impregnated or coated with a Ni compd., at an elevated temp., 500-600°) successively to the action of H and CO<sub>2</sub> and finally cooling it in an atm. of CO<sub>2</sub>.
- U. S., 1,001,290, Aug. 22. W. E. McKee, Bisbee, Ariz. Steam generator, utilizing molten slag, the admission of which into the H<sub>2</sub>O is automatically controlled.
- U. S., 1,001,466, Aug. 22. E. E. SLAUGHTER, Clifton, Ariz. Extracting copper from weak solutions by immersing in the soln. a number of small pieces of Fe and C (preferably coke) closely packed together and treating the resulting mass in a blast furnace to obtain metallic Cu.
- U. S., 1,001,525, Aug. 22. G. P. Hulst, Omaha, Nebr. Refining and desilvering impure lead bullion by m. it, crystg. a portion of the Pb from the molten bullion by cooling and agitation, sepg. the molten impure Pb from the cryst. Pb by mechanical pressure applied to the crystals and conducting the impure Pb away from the crystals.
- U. S., 1,001,536, Aug. 22. S. LILJA, Bayonne, N. J. Reducing hematite to magnetic iron oxide by heating with a S compd. of Fe, c. g., pyrites or pyrrhotite.
- U. S., 1,001,669, Aug. 29. J. F. Monnot, New York, N. Y. Forming compound metal objects by applying Cu in a supermolten state to a ferrous metal base to form

- a layer of Cu having a cohering union with the base, extending the billet so formed, placing a layer of Ag or Au on one side of the extended billet and working the metals to unite them while hot.
- U. S., 1,002,018, Aug. 29. C. H. WRIGHT, Cleveland, Ohio. Apparatus for charging blast furnaces.
- U. S., 1,002,037, Aug. 29. F. L. CLERC, Boulder, Colo. Reducing zinc oxide and recovering the resulting Zn vapors; by establishing a trapped body of substantially quiescent molten metal in the bottom of a retort, the trapped body terminating outside the retort in an outer well, supplying a charge of ZnO and C to the surface of the trapped portion within the retort, heating the metal in the outer well, and reducing the ZnO by the C of the charge by means of the heat conducted from the well, through the molten metal to the surface of the trapped portion within the retort.
- U. S., 1,002,133, Aug. 29. G. BRYANT, Finsbury Park, London, and C. H. IVINSON, Ilford, Eng. Treating metals to render them more easy to alloy, e. g., in making solder, by bringing the finely divided molten metals, e. g., Zn or Sn, into intimate contact with successive layers of oil containing a cleaning reagent, e. g., P, S or I, and H<sub>2</sub>O containing ZnCl, or NH<sub>4</sub>Cl.
- U. S., 1,002,190, Aug. 29. W. WYERS, Norristown, Pa. Antifriction alloy containing Zn 78%, Cu 12%, Pb 7%, Fe 2% and Mn 1%.
- U. S., 1,002,446, Sept. 5. J. E. PORTER, Syracuse, N. Y. Assignor to Just Mining and Extraction Co., Rochester, N. Y. Recovering precious metals from ores by maintaining the fine ore in suspension in a cyanide soln. by the introduction thereinto of minute air bubbles, and while so suspended sepg. the values from the soln. upon surfaces of Al.
- U. S., 1,002,447, Sept. 5. Idem. Treating refractory sulfide ores containing gold and silver by maintaining the ore previously reduced to a state of extremely fine subdivision in suspension in a heated cyanide soln., and distributing air in minute subdivision through the soln. to maintain oxidizing conditions therein, the conditions being so adjusted that the S of the ore is converted into thiosulfate.
- U. S., 1,002,577, Sept. 5. J. GAYLEY, New York, N. Y. Drying air by passing it through a cooling tower wherein it is subjected to the action of artificially cooled H<sub>2</sub>O at a temp. of about 2°, then passing it at a speed no greater than in the cooling tower through a refrigerating chamber of greater capacity than the direct-action tower and reducing the moisture therein to less than 2.5 grains per cu. ft. by reducing the temp. of the air to or below o°, the dried air being then fed to a metallurgical app.
- U. S., 1,002,578, Sept. 5. Idem. Drying air for metallurgical purposes by passing it upwardly through a chamber and subjecting it therein to the direct action of a descending shower or spray of artificially cooled H<sub>2</sub>O, refrigerating the H<sub>2</sub>O out of contact with the air being treated and thereafter passing the air upwardly through another chamber or compartment, subjecting it therein to the direct action of a cooling liquid (e. g., brine) to reduce its temp. to or below o° to reduce the moisture content to less than 2.5 grains per cu. ft., the air being then used in a metallurgical app.
- U. S., 1,002,698, Sept. 5. E. Jonassen and F. Pytterud, Christiania, Norway. Pulp-refining apparatus.
- U. S., 1,002,865, Sept. 12. F. I. DU PONT, Wilmington, Del. Concentrating iron ores; see C. A., 5, 3042.
- U. S., 1,002,889, Sept. 12. H. L. WHITTEMORE, Urbana, Ill. Casting metal, by first carving an intaglio of the article to be cast in a block of paraffin wax, filling the intaglio with a concrete of hydraulic cement in fluid or plastic condition, causing

- the concrete to set to form a hard pattern, removing the pattern from the wax intaglio, surrounding the pattern with tamped molding sand in a suitable flask, drawing the pattern from the sand mold, closing the latter and pouring in the metal to be cast.
- U. S., 1,003,051, Sept. 12. D. KORDA, Paris, France. Assignor to Soc. Com. des Mines, Min. et Met., Paris, France. Treating sulfuretted copper and zinc ores rich in zinc by mixing the ore with coke in small pieces, m. the mixt. in a converter formed with a lining containing a high proportion of coke, injecting compressed air into the molten metal, and prior to swinging the converter adding to the molten mass a small quantity of pyrites containing very little Cu and Zn, to remove the Cu content from the slag and unite it to mat already formed.
  - U S., 1,003,118, Sept. 12. C. McKellar, Chicago, Ill. Amalgamator.
- U. S., 1,003,275, Sept. 12. N. Kostillev, Nischne Saldinskij Sawod, Russia. In casting steel ingots, placing a head box upon the ingot mold, pouring liquid metal into the mold and head box, removing the head box while the metal is still in viscous condition and pouring cold H<sub>2</sub>O on top of the metal.
- U. S., 1,002,401, Sept. 5. W. HOMMEL, London, Eng. Assignor to Metals Extraction Corporation, Ltd., same place. Recovering zinc from waste zinc furnace distillation residues containing carbonaceous matter, and simultaneously extracting Zn from fresh Zn ore, by roasting the ore, mixing it with the Zn residues containing sufficient C to effect the reduction of all the Zn in the mixt., igniting the mixt., so as to blow off the Zn (which burns to form ZnO) in admixt. with other metallic matter, collecting the volatilized product and treating it with a soln. of SO<sub>2</sub> so as to obtain a soln. of Zn(HSO<sub>2</sub>)<sub>2</sub>, and pptg. the Zn as insol. ZnSO<sub>3</sub> which is then calcined to produce ZnO.
- U. S., 1,003,406, Sept. 12. H. OTTMANN, Munich, Ger. Treating blast furnace slag from a furnace working the gray-iron process, to blow or puff up the slag, by introducing H<sub>4</sub>O into the interior of a coherent mass of the molten slag.
- U. S., 1,002,453, Sept. 5. A. RONAY, Berlin, Ger. Assignor to Allg. Briketierungs-G. m. b. H., Berlin, Ger. Briquetting and pressing waste metal without the aid of an agglutinant by subjecting the waste to a very high pressure which rizes during a definit fraction of the duration of the pressure an increasing amt. per unit of time as the % of air in the material decreases.
- U. S., 1,003,595, Sept. 19. C. GIESECKE, Brunswick, Ger. Apparatus for dressing ores containing gold and silver.
- U. S., 1,003,627, Sept. 19. W. MATHESIUS, Charlottenburg, Ger. Reducing metal from metallic oxide ores, e. g., of Fe; (see C. A., 5, 3040).
- U. S., 1,003,628, Sept. 19. W. MATHESIUS, Charlottenburg, Ger. Apparatus for treating solids with gases,  $\epsilon$ . g., in the reduction of ores of Fe, Co, Ni, Cu, Sn, etc.
- U. S., 1,003,682, Sept. 19. F. W. Yost, Chicago, Ill. Treating with gases finely divided material containing gas-forming components to secure the action of the introduced gas without its previous contamination by initiating a reaction at one surface of a mass of the material having definit bounding surfaces and capable of undergoing a propagative reaction, e. g., in roasting sulfide ores, the mass being of substantially uniform character throughout, introducing a reaction-supporting gas, e. g., air, at another surface of the mass and conducting it through the interstices of the material, controlling the conditions so as to cause an internal propagation of the reaction throughout the body of the mass, and withdrawing the volatil products of reaction at the surface of initial reaction.
  - U. S., 1,003,683, Sept. 19. Idem. Treating finely divided material containing

- gas-forming constituents,  $\epsilon$ . g., in clinkering oxide ore mixed with coal or coke, by initiating a reaction at a surface of a mass of the material, conducting a reaction-supporting gas,  $\epsilon$ . g., air, through the mass toward the region of initial reaction, propagating the initial reaction through the mass in one direction and propagating a second reaction through the mass in another direction,  $\epsilon$ . g., by causing the clinkering to take place progressively from bottom to top after combustion in the charge has progressed from top to bottom.
- U. S., 1,003,684, Sept. 19. Idem. Treating finely divided material containing gasforming components, e. g., oxide ores, mixed with fuel, by heating the upper surface
  of a mass of the material so as to initiate a clinkering reaction at that surface, introducing a combustion-supporting gas, e. g., air, at the lower surface and conducting it
  upward through the mass, propagating the heat downward through the mass from the
  upper to the lower surface by internal combustion to clinker the upper portion of the
  mass, and propagating a clinkering reaction upward from the lower surface to clinker
  the lower portion of the mass.
- U. S., 1,003,704, Sept. 19. T. CHARLTON, Chicago, Ill. Apparatus for separating constituents of ores having different specific gravities, by settling in chambers filled with quiescent liquid.
- U. S., 1,003,781, Sept. 19. F. Ondra, Ferreira, Johannesburg, Transvaal. Apparatus for precipitating metals from solution, comprizing 2 vessels, a common receiver into which both vessels open at the bottom, connections for passing liquid into the upper part of one vessel and out of the upper part of the other, connections for supplying a precipitant gas under pressure and distributing it in the lower part of the vessel into which the soln. first flows and connections for withdrawing gas from the top of the vessel and for controlling the outflow of liquid from the bottom and sepn. of the ppt. therefrom.
- U. S., 1,003 805, Sept. 19. A. J. Rossi, Niagara Falls, N. Y. Treating steel by incorporating therewith while molten a quantity of an alloy of Fe with Ti, the Ti being proportioned to combine with undesired substances, e. g., O, N and oxides, and produce a final product containing substantially no Ti.
- U. S., 1,003,806, Sept. 19. *Idem*. Alloy of titanium with nickel, made by incorporating TiO<sub>2</sub> into a bath of molten Ni and Al, subjecting the bath to a temp. sufficient to insure reduction of the TiO<sub>2</sub> by the Al and withdrawing and cooling the resulting metallic product.
- Brit., 14,276, June 11, 1910. F. W. Yosr, 135 Adams St., Chicago, Ill. Clinkering, roasting calcining, or otherwise treating materials by treating on a perforated hearth or its equiv., other than a hearth formed of porous material, only material which is finely divided and which naturally contains or which is artificially and intimately mixed with heat developing components or gas forming components, forming a mass of the material or mixt. wherein the heat developing components or gas forming components are distributed in all parts of the mass with substantial uniformity and wherein interstices are distributed through all parts of the mass for the substantially uniform passage of a gas current through all parts of the mass, initiating a reaction at a surface of the mass, introducing a reaction-supporting gas at another surface, conducting the reaction-supporting gas through the interstices, and controlling conditions so as to cause propagation of the reaction through the mass.
- Brit., 11,832, May 12, 1910. WM. S. SIMPSON, 49 Battersea Park Road, London. In app. for the melting, mixing, and heat treatment of metals, the employment of the resistance of a crucible made of refractory or carbonaceous material to the passage of an elec. current which is applied to the top and bottom of such crucible and pro-

vides a direct means of generating the heat required; such crucible being suitable for m, metals and being enclosed in a vacuum chamber.

Brit., 11,869, May 13, 1910. G. J. STOCK, Cockerton Hall, Darlington. In Bessemer converters, the construction and arrangement of app. or combination of parts such that a sep. furnace for m. and a sep. converter for refining air used in conjunction with an economizer for heating the air whereby the m. furnace is adapted to be worked continuously and both m. furnace and converter work economically by using air heated by the products of combustion which escape from the m. furnace.

Brit., 21,206, Sept. 12, 1910. J. HARDEN, et al., 9 Porthminster Terrace, St. Ives. In the production of tin from its ores by reduction, effecting the production in a single furnace and at a high temp. such that by adding to the furnace that contains the impure Sn resulting from the reduction, a predetd. amt. of a strong and rapid oxidizing agent, the combined action of this agent and of the high temp. causes the removal of the impurities without, however, materially affecting the Sn.

#### 10. ORGANIC CHEMISTRY.

#### I. BISHOP TINGLE.

State of Substances in Solution in Absolute Sulfuric Acid. A. HANTZSCH. Gass. chim. ital., 41, I, 645-66. Refutation of the criticisms of Oddo and Scandola (C. A., 5, 1744).

Amino Alcohols and Derivatives with Therapeutic Properties. II. E. FOURNEAU.

J. pharm. chim., [7] 2, 109-17; cf. C. A., 5, 1971.—Dimethylaminodimethylethylcarbinol,
Me<sub>2</sub>NCH<sub>2</sub>CMeEtOH, (1) by heating C<sub>2</sub>H<sub>6</sub> soln. of Me<sub>2</sub>NH with ClCH<sub>2</sub>MeCEtOH in a
sealed tube, or (2) from Me<sub>2</sub>NH and EtMeC — CH<sub>2</sub> in the same way, or (3) by the

action of EtMgBr on Me<sub>2</sub>NCH<sub>2</sub>Ac; colorless liquid b. 149°, methiodide, prisms, m. 126°. The other bases described were prepared similarly. Diethylaminodimethylethylcarbinol. Dimethylaminotrimethylcarbinol, b<sub>788</sub> 130°. Methiodide, prisms, m. 130°. Dimethylaminodimethylpropylcarbinol, b. 78°. Methiodide, plates, m. 151°. Dimethylaminodimethylisoamylcarbinol,  $b_{ns}$  199.5°. Hydrochloride, leaslets, m. 145°. Dimethylaminodimethylphenylcarbinol. Dimethylaminodimethylbenzylcarbinol,  $b_{mi}$  144°. Dimethylaminodiethylmethylcarbinol, from EtMgBr and Me,NCH,CO,Et, or EtMgBr and CICH, CO, Et, followed by Me, NH, b. 170°. Methiodide, prisms, m. 134°. Starting with excess of NH<sub>2</sub>, by method (1) both primary and secondary bases are obtained. Aminodimethylethylcarbinol, HaNCHaCMeEtOH, syrup with nicotine odor, b. 170°, not pptd. by the usual alkaloid reagents. Hydrochloride, hygroscopic leaflets, m. 90°. Aminobisdimethylethylcarbinol, syrup with strong odor, by 165-6°. Hydrochloride, brilliant, non-hygroscopic leaflets. Aminodimethylisoamylcarbinol, bea 125°. Aminobisdimethylisoamylcarbinol, cryst., bus 206°. Hydrochloride, difficultly sol. in H2O, affording a sepn. from the primary base, m. 183°. Aminodimethylphenylcarbinol, b40 174-5°. Aminobisdimethylphenylcarbinol, b40 258°. Hydrochloride, slightly sol. in H<sub>2</sub>O, leaflets, m. 228-9°. Methylaminodimethylethylcarbinol, b. 160°.

E. J. WITZEMANN.

Derivatives of Amino Alcohols. I. Esters or Derivatives Acylated at the Oxygen. E. FOURNEAU. J. pharm. chim., [7] 2, 337-44; cf. preceding abstract.—The esters of amino alcs. were prepared by mixing C<sub>0</sub>H<sub>6</sub> solns. of the alcs. and the acid chlorides or bromides; the ester salts crystallize out almost immediately. With certain amino alcs. like diethylamino alcohol it was necessary to use no solvent in order to obtain the benzoate. Amino alcs. in which both functions are tertiary form esters with

ClCO<sub>2</sub>Et, as with other acid chlorides, differing thus from the simple tertiary alcs. In this case the tertiary NH, acts like pyridine in acylation. The amino alc. esters are nearly odorless liquids, which are more easily hydrolyzed by strong mineral acids than by alkalies. Derivs. of dimethylaminotrimethylcarbinol, Me,NCH,CMe,OH: Benzoate hydrochloride, cubes, m. 202°, cinnamate hydrochloride, large plates, m. 208°, isovalerate hydrochloride, hygroscopic, unctuous leaflets, m. 125°. Derivs. of dimethylaminodimethylethylcarbinol: Benzoate, syrupy liquid, b, 150°, benzoate hydrochloride is stovaine, diethylcarbamate derivative, Me2NCH2C(O2CNEt2)MeEt, limpid liquid with peculiar odor ba 136°. Hydrochloride, hygroscopic needles from acetone, m. 142° (decomp.), hydrobromide, sedative, needles, m. 148°, chloroaurate, long orangered needles m. 98°, "valerate" derivative, b, 128°, very stable to alkalies, hydrochloride, anesthetic, hygroscopic, unctuous plates, m. 151°, hydrobromide, similar, but not hygroscopic, m. 126°. Bromovalerate hydrochloride, strongly anesthetic, fine needles, m. 158°. Diethylacetate hydrobromide, brilliant needles, m. 169°. Bromodiethylacetate hydrochloride, leaflets, m. 160°. Caproate derivative, b. (in vacuo) 152°. Hydrochloride, slender needles. The compds. with the aliphatic acids heavier than caproic are strongly modified by the acyl group and acquire the properties of soaps like the polypeptides of the higher aliphatic acids. Bromocaproate hydrochloride, slender hygroscopic needles, m. 130°. Aq. soln. gives lather. Bromo-oenanthylate hydrochloride, white leaflets, m. 128° anesthetic. Bromolaurate hydrochloride, slender needles, m. 99°. Aq. soln. gives lather. Dimethylaminomethyldiethylcarbinyl benzoate hydrochloride, Et.C(OBz)CH.NMe, HCl, quadrangular plates, m. 180°. Dimethylaminodimethylpropylcarbinyl benzoate hydrochloride, isomeric with the preceding compd., brilliant needles, m. 146°. Dimethylaminodimethylisoamylcarbinyl benzoate hydrochloride, anesthetic, long needles, m. 142°. Chloroplatinate, m. 178°. Cinnamate hydrochloride, slender needles, m. 110°. Diethylaminodimethylethylcarbinyl benzoate hydrochloride, tufts of slender needles, m. 140°. II. Derivatives Acylated at the Nitrogen. Ibid., [7] 2, 397-401.—These compds. were prepared by the Schotten-Baumann method from the amino alcs. Bromoisovalerylaminodimethylethylcarbinol, Me\_CHBrCHCONHCH\_CMeEtOH, large transparent octahedral crystals, m. 93°. "Valeryl" aminodimethylethylcarbinol, rhombic plates, m. 50-60°, b. 190°. The -OH is not attacked, even in the presence of an excess of the acid chloride. "Valeryl" aminobisdimethylethylcarbinol, leaflets, m. 152-3°, bm 210°. These compds. are strongly sedative in their action, even hypnotic, and are less toxic than the amino alc. esters. Carbethoxyaminodimethylethylcarbinol, EtO, CNHCH, CMeEtOH, syrup, hypnotic and diuretic, b<sub>17</sub> 151-2°. Carbopropoxyaminodimethylethylcarbinol, PrO<sub>2</sub>CNHCH<sub>2</sub>C-MeEtOH, syrup, b<sub>22</sub> 174-5°. Uraminodimethylethylcarbinol, m. 150°. Methyluraminodimethylethylcarbinol, m. 128°. Uraminobisdimethylethylcarbinol, leaflets, m. about 90°. The urethans and carbamides are hypnotic only in large doses, showing the antagonistic action of the -OH. E. J. WITZEMANN.

Oxidation of Tertiary Alcohols of the Tolylallylic Series. E. GRISHKEVICH-TROK-HIMOVSKII. Univ. for Women, Kiev. J. Russ. Phys. Chem. Soc., 42, 1543-52; cf. C. A., 4, 2439.—The oxidation of the alcs. with a 40% KMnO<sub>4</sub> soln. gave glycerols and hydroxy acids, which could be extracted separately with Et<sub>2</sub>O. The structure of the acids was proved by means of their dry distillation, corresponding hydrocarbons being the result. The following alcs. have been investigated: (1) Me-p-tolylallylcarbinol, (2) Et-p-tolylallylcarbinol, (3) Pr-p-tolylallylcarbinol, and (4) iso Pr-p-tolylallylcarbinol. The glycerols obtained were (1) 2-p-tolylpentane-2,4,5-triol, MeCh<sub>4</sub>CMe(OH)CH<sub>2</sub>CH(OH)CH<sub>2</sub>OH m. 101-103°, needles. (2) 3-p-Tolylhexane-3,5,6-triol, MeCh<sub>2</sub>Ch<sub>4</sub>CMe(OH)CH<sub>2</sub>CH(OH)CH<sub>2</sub>OH m. 89-90.5°, needles. (3) 4-p-Tolylheptane-4,6,7-triol, MeCh<sub>2</sub>Ch<sub>4</sub>CMe(OH)CH<sub>2</sub>CH(OH)CH<sub>2</sub>CH(OH)CH<sub>2</sub>CH(OH)CH<sub>2</sub>OH thick liquid. (4)

2-Methyl-3-p-tolylhexans-3,5,6-triol, MeCHMeC<sub>2</sub>H<sub>4</sub>CMe(OH)CH<sub>2</sub>CH(OH)CH<sub>2</sub>OH, very thick liquid. The hydroxy acids, in the same succession, were: (1)  $\beta$ -p-Tolylmethylene-lactic acid, MeC<sub>4</sub>H<sub>4</sub>CMe(OH)CH<sub>2</sub>CO<sub>2</sub>H, m. 103-106°, needles from Et<sub>2</sub>O, (2)  $\beta$ -Et-p-Tolylethylenelactic acid, m. 109-111°, (3)  $\beta$ -Pr-p-tolylethylenelactic acid, m. 99-101°, needles from Et<sub>2</sub>O, (4)  $\beta$ -iso Pr-p-tolylethylenelactic acid, m. 106-108°, needles. The corresponding hydrocarbons were: (1) Me-p-tolylethylene, MeC<sub>4</sub>H<sub>4</sub>CMe: CH<sub>2</sub> b<sub>748</sub> 186-9°, d<sub>4</sub><sup>23</sup> 0.8936, colorless liquid, adding Br readily, (2) Et-p-tolylethylene, b<sub>749</sub> 206-209°, d<sub>4</sub><sup>22.5</sup> 0.8926; n<sub>4</sub><sup>22.5</sup> 1.52735, (3) Pr-p-tolylethylene C<sub>12</sub>H<sub>16</sub> b. 221-4°, (4) isopropyl-p-tolylethylene, C<sub>12</sub>H<sub>16</sub> b. 210-212°, d<sub>4</sub><sup>27</sup> 0.8838. G. Weintraub.

Study of Hydroaromatic Substances. Report of a Committee. Chem. News, 104, 141-2.

T. E. LAYNG.

Transformation of Aromatic Nitroamines and Allied Substances and its Relation to Substitution in Benzene Derivatives. Report of a Committee. Chem. News, 104, 144-5.

T. E. L.

Stereoisomeric Chloroimino Ketones. P. P. PHTERSON. Kent Chem. Lab., Univ. Chicago. Am. Chem. J., 46, 325-44.—At  $0^{\circ}$  p-chlorobenzophenoneimine is converted by HClO (2 mols.) into  $\alpha$ - and  $\beta$ -p-chlorobenzophenonechloroimine, formulas (I) and (II), resp. Total yield, 80%. They are sepd. by adding ligroin to the CHCl<sub>8</sub>

soln., which ppt. the  $\alpha$ -form in thin plates m. 104°,  $\beta$ -deriv., thin prisms or needles, m. 55°. In ligroin each isomer is converted by dry HCl, followed by H<sub>2</sub>O, into  $\beta$ -chlorobenzophenone. The  $\alpha$ -form, by successive treatment with HCl and HClO, is transformed into a mixt. of  $\alpha$ - and  $\beta$ -isomers. After m. each form is recovered unchanged and from solns. to which a crystal of the isomeric substance has been added, the original material is deposited unchanged. Corresponding compds. of  $\beta$ -methoxy-bensophenons have been prepared;  $\alpha$ -form thin hexagonal plates m. 90°;  $\beta$ -form large asymmetric crystals m. 54°; mixt. of  $\alpha$ - and  $\beta$ -forms m. 40°. Also compounds of  $\beta$ -chloro- $\beta$ -methoxy-bensophenone;  $\alpha$ -form m. 94°;  $\beta$ -form m. 64°: mixt. m. 50–52°. These have been prepared in a similar manner and have similar reactions and transformations. The higher m.  $\alpha$ -form of these has the simple mol. wt. Unsuccessful attempts were made to prepare compds. of  $\beta$ -nitrobenzophenone and of  $\beta$ -methoxy-o'-chlorobenzophenone. The above facts lend support to Stieglitz's explanation of the Beckmann rearrangement.

Researches on Pyrimidines. CXCIII. The Condensation of Ethyl Formate and Diethyl Oxalate with some Pyrimidine-thioglycolates. TREAT B. JOHNSON AND NORMAN SHEPARD. Sheffield Lab., Yale Univ. Am. Chem. J., 46, 345-61.—Ethyl 6-oxypyrimidine-2-thioglycolate (I), was prepared by the action of Et chloroacetate on thiouracil in presence of an alkali. There is also formed a cryst. compd. which was septd. from NaCl by its insolubility in H<sub>2</sub>O. It is probably trans-β-thioureaccrylic acid because of its stability in presence of HCl. When the Na salt of (I) is treated

with HCO<sub>2</sub>Et, in presence of Na, ethyl 6-oxypyrimidine-2-(a-thio-\(\theta\)-hydroxyacrylate) (II), well-developed prisms, m. 138-140° results. When (II) is digested with conc. HCl it forms 6-oxypyrimidine-2-thiopyruvic acid (III), colorless crystals

from alc. m. 200-1°. Uracil is formed from (II) and KOH. The Na salt of (I) produces with thiourea in presence of HgCl, and H<sub>2</sub>S, 2-(2-thio-6-oxypyrimidine-

5-mercapto)-6-oxypyrimidine (IV) needles from H<sub>2</sub>O, m. 285-95°. The Na salt of (II), when treated with thiourea in presence of HgCl<sub>2</sub> and H<sub>2</sub>S produces a salt very similar to that produced by the action of thiourea on (I), needle-like prisms from alc. m. 290-4°, not definitly identified. 2-Thio-4-methyl-6-oxypyrimidine was prepared by condensing thiourea with Et acetoacetate in alc. Ethyl 4-methyl-6-oxypyrimidine-2-thioglycolate (V) was prepared by the action of the Na oxypyrimide on Et chloroacetate, colorless needles from alc. m. 145-6°. When (V) is treated with

alc. KOH the dipotassium salt results; with excess of HCl 4-methyl-6-oxypyrimidino-z-thioglycolic acid (VI), colorless prisms from H<sub>2</sub>O, m. 192-7° results. The Na salt of (V) when treated with HCO<sub>2</sub>Et (2 mols.) produces ethyl 4-methyl-6-oxypyrimidine-z-( $\alpha$ -thio- $\beta$ -hydroxyacrylate) (VII), colorless prisms, m. 106°. A part of the yellow salt

of (V) was treated with dil. HCl producing 4-methyluracil, m. 290°, with the pyrimidine. The attempt to reduce (VII) with Na-Hg produces 4-methyl-6-oxypyrimidine-2-thioglycolic acid (VIII), colorless prisms from MeCO<sub>2</sub>H, m. 192-3°. The action of thiourea on (VII) in presence of HgCl<sub>2</sub> and H<sub>2</sub>S produced 2-(2-thio-6-oxypyrimidine-5-mercapto)-4-methyl-6-oxypyrimidine (IX). Clusters of prisms m. above 300°. The

action of Et oxalate in presence of NaOEt on (V) produced diethyl 4-methyl-6-oxypyrimidine-2-oxalthioglycolate (X), stout blocks, m. 139-40°. The action of thiourea

on the Na salt of (X) in presence of HgCl<sub>2</sub> and H<sub>2</sub>S produces a cryst. substance; hexagonal tables m. 164-5° probably (XI) or (XII).

T. E. LAYNG.

Action of Carbon Tetrabromide on Organic Bases. WILLIAM M. DEHN AND ALBERT H. DEWEY. Univ. Washington. J. Am. Chem. Soc., 33, 1588-98.—On mixing equimol. quantities of piperidine and CBr<sub>4</sub> in dry Et<sub>2</sub>O soln., piperidine dicarbontetrabromide, C<sub>2</sub>H<sub>11</sub>N(CBr<sub>4</sub>)<sub>2</sub>, prismatic needles, m. 148°, is formed. When heated it yields C<sub>2</sub>H<sub>11</sub>NCBr<sub>4</sub>, C<sub>2</sub>H<sub>11</sub>N and CBr<sub>4</sub>; in presence of H<sub>2</sub>O it forms C<sub>4</sub>H<sub>11</sub>N.HBr, CBr<sub>4</sub>, HBr and CO<sub>2</sub>. Bromoaurate, C<sub>4</sub>H<sub>11</sub>N.HAuBr<sub>4</sub>, dark, red-brown needles, m. 242°. Sunlight greatly accelerates the reaction with pyridine. Pyridine dicarbontetrabromide, iridescent crystals from CHCl<sub>2</sub>, darkens 155° m. 218-20°. When heated this compd.

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loses I mol. CBr<sub>4</sub> below 100° and the 2nd mol. near its m. p. Bromoaurate, fine brown prismatic needles, m. 319°. Quinoline carbontetrabromide, white needles from CHCl<sub>2</sub>, softens 110°, m. 142°. Its H<sub>2</sub>O soln. on evap. yields quinoline hydrobromide, white needles m. 62-5°. Bromoaurate, C<sub>2</sub>H<sub>7</sub>N.HAuBr<sub>4</sub>, small, dark brown needles, m. 209°. α-Picoline dicarbontetrabromide, large needles from CHCl<sub>2</sub>, soften 190°, m. 214°. Picoline bromoaurate short, dark-red prisms m. 209°. With lutidine, crystals m. 106° were obtained. Lutidine hydrobromide, white needles, m. 114°. Bromoaurate, dark brown needles, m. 190°. With PhN<sub>2</sub>H<sub>3</sub>, the hydrobromide is formed and N evolved. Benzylamine yielded the hydrobromide; bromoaurate, long brownish red flakes, m. 178°. With diisoamylamine and dipropylamine crystals were obtained, but their comp. was not definit. Ethylamine hydrobromide, white, feathery crystals, m. 157°. Bromoaurate, coarse, red-brown prisms m. 190°, ethylamine also yielded a compd. m. 150°. The mechanism of the above reactions is discussed.

STUART J. BATES.

Action of Diiodoacetylene on Organic Bases. WILLIAM M. DEHN. Univ. of Washington. J. Am. Chem. Soc., 33, 1598-1601.—Diiodoacetylene is best prepared by conducting C<sub>2</sub>H<sub>2</sub> into a soln. of KI to which a soln. of NaClO or NaBrO is being slowly added. In Et<sub>2</sub>O soln. it unites with Et<sub>2</sub>N forming triethylaminediiodoacetylene, Et<sub>2</sub>N(C<sub>2</sub>I<sub>2</sub>)<sub>2</sub>, light yellow crystals, darken 97°, m. 115°. Dipropylaminediiodoacetylene, long needles m. 160°. Piperidinediiodoacetylene, colorless needles darken 117°, m. 161°. PhN<sub>2</sub>H<sub>2</sub> and diiodoacetylene yield (a) diphenylhydrazine hydriodide, (PhN<sub>2</sub>H<sub>2</sub>)<sub>2</sub>.HI, m. 128° (decomp.) (b) phenylhydrazine hydriodide, PhN<sub>2</sub>H<sub>2</sub>.HI, darkens 270°, not m. 500°, (c) phenylacetyleneiodide, and (d) N.

New Method for Obtaining Amines, Using Grignard's Reaction. BENITO BUYLLA. Univ. Bologna, Lab. of Prof. Ciamician. Rev. real. acad. cien., Madrid, 9, 635-53, 718-34.—An attempt to obtain Mg: NEt (and from this the tertiary amine) by the action of Mg and dichloroethylamine in Et<sub>2</sub>O soln. did not succeed. The mixture, which reacted only with Mg powder strongly heated with I, produced MeCN and HCl. An attempt to obtain Et<sub>2</sub>N by the action of iododiethylamine and EtMgBr in Et<sub>2</sub>O also failed, the iododiethylamine being recovered quant. Et<sub>2</sub>N from EtMgBr and Et<sub>2</sub>NH followed by EtBr, the reaction being completed in b. xylene. With PhMgBr at 210° during 4 days as above a trace of Ph<sub>2</sub>NH was obtained. H. S. Paine.

Halogenated Derivatives of Anthraquinone. José Prats Aymerica. Anales soc. españ. fis. quim., 9, 54-61; Rev. real. acad. cien., Madrid, 9, 442-50.—Discussion of the prep. of  $\alpha$ -chloro-,  $\beta$ -chloro-,  $\alpha$ -bromo-, 1,5-dichloro-, 1,8-dichloro-, tetrabromo-, and heptabromoanthraquinone by Sandmeyer's reaction, direct substitution, oxidation of corresponding halogenated anthracene derivs., replacement of SO<sub>2</sub>H in sulfonated derivs., or from chloro- or bromobenzoylbenzoic acids. H. S. Paine.

Relation between Chrysophanic Acid, Aloe-emodin and Rhein. O. A. OESTERLE. Arch. Pharm., 249, 445-9.—Aloe-emodin on reduction gives chrysophanic acid and on oxidation rhein, and chrysophanic acid on oxidation gives rhein. The groupings are as follows:

H. C. FULLER.

Action of Organo-magnesium Compounds on 4- Methoxyphthalic Anhydride.

H. BAUER. Arch. Pharm., 249, 450-3.—The configuration of diethylphthalide has been determined through nitration and subsequently treating the hydrazo deriv. with mineral acids, and is now substantiated by the action of EtMgBr on 4-methoxyphthalic anhydride. This might produce 4- or 5-methoxydiethylphthalide, the latter being identical with the same compd. obtained through the nitration of diethylphthalide, while the former on fusion with KOH would give diethyl ketone and anisic acid. It has been detd. that the action of organo Mg compds. is on the CO m- to the MeO. This would be the case when 4-methoxydiethylphthalide results. The compd. obtained above did not react as a 4-MeO derivative and furthermore on treatment with NHO<sub>3</sub> only one mononitro deriv. was obtained and no dinitro deriv.

Action of Organo-Magnesium Compounds on Homophthalic Anhydride. H. BAUM AND E. WOLZ. Arch. Pharm., 249, 454-8.—A simple reaction for preparing dialkyl and diaryl phthalides was obtained by the action of organo Mg compds. on benzaldicarbonic carboxylic anhydride. At the same time other reactions were noted; with PhMgBr or with p-tolyl MgBr, an orthodiketone resulted at the same time as the phthalide, and with certain substituted phthalic anhydrides, monoalkylphthalides resulted, the reaction not being entirely clear. These anomalies led to the study of the reaction with homophthalic anhydride. On adding it to a soln. of the Mg compd. in Et<sub>4</sub>O a mild reaction took place giving a dialkyl and a diarylphthalide. The Mg compd. might give (I) or (II). The dialkylphthalides on fusion with KOH yielded

dialkyl ketones and BzOH and this reaction should give with dimethylhomophthalide in the case of (I) Me<sub>2</sub>CO and phenylacetic acid, and in the case of (II) Me<sub>2</sub>CO and o-toluic acid, but the reaction did not take place in either of these directions. H. C. F.

Use of the Magnetic Field as a Test of Constitution. X. P. PASCAL. Fac. sci. Lille. Bull. soc. chim., 9, 809–12; cf. C. A., 5, 2631.—While liquid compds. capable of existing in tautomeric forms show a magnetic susceptibility intermediate between the calc. values for the keto and enol forms, resp., solid compds. give values equal to those calc. for the one or other form, and this const. can therefore be used as a test for the constitution of such compds. The % of enol form in a variety of substances was found by this method to be as follows: Fresh AcCH<sub>2</sub>CO<sub>2</sub>Et, 67; old, 17; AcCHMeCO<sub>2</sub>Et, 0; AcCHEtCO<sub>2</sub>Et, 0; Me<sub>2</sub>CHCH<sub>2</sub>CHAcCO<sub>2</sub>Et, 0; BzCH<sub>2</sub>CO<sub>2</sub>Et, 63; AcCH<sub>2</sub>Ac, 63.5; CO(CH<sub>2</sub>Ac)<sub>2</sub>, 36.5; AcCHMeAc, 15; cyclohexanone, 78; o-methyl-cyclohexanone, 38; m-methylcyclohexanone, 52; m-C<sub>6</sub>H<sub>4</sub>(OH)<sub>2</sub>, 100; 1,3,5-C<sub>6</sub>H<sub>9</sub>(OH)<sub>4</sub>, 100; carvacrol, 100; carvol, 100; quinone, 2; triquinoyl 98; quinonoxime, 0; dinitroresorcinol, 0.

p-Hydroxybenzylamine. M. TIFFENEAU. Bull. soc. chim., 9, 819-24.—p-MeOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>NH<sub>2</sub>, b<sub>14</sub> 122-4°, b<sub>700</sub> 236-7°, d<sub>16</sub> 1.050; hydrochloride, m. 240-1; hydroiodide, m. 183-4°. Heated with conc. HI, the base gives p-HOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>NH<sub>2</sub>.HI, m. 198-200°; hydrochloride, m. 195°. p-Methoxybenzyldihydroisoindole, C<sub>6</sub>H<sub>4</sub>(CH<sub>2</sub>)<sub>3</sub>-NCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>OMe, from o-C<sub>6</sub>H<sub>4</sub>(CH<sub>2</sub>Br)<sub>2</sub> and p-MeOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>NH<sub>2</sub> in abs. alc., needles, m. 83°; hydrobromide, scales, m. 234°; methiodide, m. 183°. Ac<sub>2</sub>O, on long b., decomp. the compd. into p-MeOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>OAc and acetyldihydroisoindole, AcN(CH<sub>2</sub>)<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, needles, m. 77°.

Chas. A. Rouiller.

Monomethyl- and Dimethyl-p-hydroxybenzylamine. M. TIFFENEAU. Bull. soc. chim., 9, 825-8; cf. preceding abstr.—p-Methoxybenzyl chloride, from the alc. and dry HCl, b<sub>18</sub> 116-20°, d<sub>0</sub> 1.072. Bromide, b<sub>6</sub> 129°, d<sub>18</sub> 1.395. Either the chloride or

bromide, mixed in a sealed tube with 20% MeNH<sub>2</sub> in alc., gives p-methoxybensylmethylamine, b<sub>14</sub> 121°, d<sub>0</sub> 1.025; hydrochloride, m. 166°; hydrocodide, m. 145°, heated with conc. HI, gives p-hydroxybensylmethylamine hydrocodide, m. 149-50°; hydrochloride, m. 188-90°. In the prep. of MeOC<sub>2</sub>H<sub>4</sub>CH<sub>2</sub>NHMe is also formed de-p-methoxybensylmethylamine, b<sub>18</sub> 223-5°, d<sub>0</sub> 1.0794. Di-p-hydroxybensylmethylamine hydrochloride, m. 197-9°. With Me<sub>2</sub>NH instead of MeNH<sub>2</sub> is obtained p-methoxybensyldimethylamine, b<sub>18</sub> 110-1°, d<sub>0</sub> 0.9878, d<sub>18</sub> 0.976; hydrochloride, m. 157°; hydrocodide, m. 145°; methiodide, m. 158°. Ac<sub>2</sub>O decomp. the base into MeOC<sub>2</sub>H<sub>4</sub>CH<sub>2</sub>OAc and AcNMe<sub>2</sub>. p-Hydroxybensyldimethylamine, m. 112°, alk. to litmus and phenolphthalein, does not appreciably color aq. FeCl<sub>2</sub>, reduces NH<sub>2</sub>-AgNO<sub>3</sub>, Millon's reagent and HI, decomp. by Ac<sub>2</sub>O into AcOC<sub>2</sub>H<sub>4</sub>CH<sub>2</sub>OAc and AcNMe<sub>2</sub>. The methiodide m. 158° (above), heated with conc. HI, gives p-hydroxybensyltrimethylammonium iodide, m. 191°; chloride, m. 98°.

Compound of Antipyrine and Ferric Chloride,  $(C_{11}H_{19}ON_2)_{\phi}$ . FeCl<sub>2</sub>.9 HCl, Obtained with Ferrous Chloride. CH. ASTRE AND J. VIDAL. Bull. soc. chim., 9, 836-9; cf. C. A., 5, 2631.—When 20 g. antipyrine and 20 cc. HCl are added to 4.76 g. FeCl<sub>2</sub> in 10 cc. of HCl (21° Bé.) and the whole placed in a stoppered flask which it completely fills, the soln. soon assumes a gooseberry-red color, and after 4 ds. K<sub>2</sub>Fe(CN)<sub>4</sub> shows the absence of ferrous salt and NH<sub>2</sub> in the presence of excess of NH<sub>4</sub>Cl ppts. all of the Fe. On conc. or the addition of a conc. acid, the color changes through yellow to greenish yellow, and finally is obtained the compound  $(C_{11}H_{19}ON_2)_{\phi}$ . FeCl<sub>2</sub>.9HCl, yellowish green crystals, partially m. 121-2°; its aq. soln. gives the usual tests for ferric salts. It is quite distinct from ferripyrine (Schuyten, Pharm. Ztg., 11, 282).

Aromatic Nitro Derivatives. II. R. Ciusa. Ist. chim. gen. univ. Bologna. Gazz. chim. ital., 41, I, 688-97; cf. C. A., 4, 1740.

C. A. R.

Basic Properties of Hydrazones. R. Ciusa. Gass. chim. ital., 41, I, 666-71; cf. C. A., 4, 1740. C. A. R.

Action of Alkaline Solutions on Trichloro Organic Compounds. G. Bressanin and G. Segré. Ist. chim. farm. tossic. r. univ. Padova. Gasz. chim. ital., 41, I, 671-4.—CCl<sub>2</sub>CMe<sub>2</sub>OH, when treated with 10% KOH, gives almost 1 mol. of CO. Me<sub>2</sub>CO is found in the reaction product. Chas. A. ROUILLER.

Action of Nascent Chlorine on Saccharin. P. Bertolo. Ist. chim. farm. r. univ. Catania. Gazz. chim. ital., 41, I, 698-705.—When saccharin is treated in hot HCl (1:2) with KClO<sub>4</sub> in small portions until it dissolves, it gives a compound to which is assigned the structure  $HO_2CC_0H_4SO(:NH)OK$ , tabular crystals, m. 285-6°, sol. in alk. carbonates with effervescence and is repptd. unchanged by mineral acids, does not lose its K on repeated crystn. from dil. HCl or  $H_2SO_4$ ; a:b:c=1.46335:1:1.79093.

Treated with BaCO<sub>2</sub> it forms a barium salt, C<sub>2</sub>H<sub>2</sub>O<sub>3</sub> Ba. 1.5 H<sub>2</sub>O<sub>4</sub>, silky

needles. The free acid is so sol. that it could not be obtained pure. When boiled with KOH, the K salt loses NH<sub>2</sub> and gives HO<sub>2</sub>CC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>K. Chas. A. ROUILLER.

Artemisin Hydrazone. P. Bertolo. Ist. chim. farm. r. univ. Catania. Gass. chim. ital., 41, I, 705-8.—Artemisin phenylhydrazone,  $C_{12}H_{12}O_2NNHPh$ , obtained by letting the ketone, in 40 pts. of 50% AcOH, and PhNHNH<sub>2</sub> stand in a dark place for I d., slightly yellow, leafy needles, m. 144-5° if heated rapidly, 221-2° if heated slowly,  $[\alpha]_D^{24}$  180°; sel. in conc. HCl or  $H_2SO_4$ , imparting to the latter a greenish color turning to red on the addition of a drop of HNO<sub>2</sub>; insol. in b. conc. KOH. Attempts to obtain from it by reduction a compd. of the type of hyposantonin (change of the —CH<sub>2</sub>CO—group to —CH: CH—) were unsuccessful. Chas. A. Roullers.

Chloroguaiacols. Temistocle Jona and G. B. Pozzi. Ist. chim. farm. tossic. (1) (2) (5)
r. univ. Pavia. Gazz. chim. ital., 41, I, 722-37.—5-Aminoguaiacol, HOC<sub>2</sub>H<sub>2</sub>(OMe)NH<sub>2</sub>, obtained by the reduction of HOC<sub>2</sub>H<sub>2</sub>(OMe)NO<sub>2</sub> with Sn and HCl, grayish crystals, m. 125-7°, gives a reddish brown color with aq. or alc. FeCl<sub>2</sub>; hydrochloride, greenish crystals. 1,5-Dibenzoyl-5-aminoguaiacol, m. 162-4°, is obtained from the base, NaOH and BzCl, while the hydrochloride, NaOAc and Ac<sub>2</sub>O form 5-acetyl-5-aminoguaiacol, m. 116-9°, and by the Sandmeyer reaction is obtained 5-chloroguaiacol, m. 161-3.5°, b<sub>700</sub> 237-9° (cor.), gives a yellow color with aq. FeCl<sub>2</sub>; benzoate, needles, m. 56-8°; acetate, leastets, m. 42-4°; ethyl ether, from the phenol, KOH and Et1, m. 49-51°. 4-Acetyl-4-aminoguaiacol, from the amino compd. and Ac<sub>2</sub>O in dil. AcOH, m. 111-3°, gives through the diazo compd. 4-chloroguaiacol, identical with the substance obtained by Peratoner and Ortoleva (Gazz. chim. ital., 1898, I, 228) from guaiacol and SO<sub>2</sub>Cl<sub>2</sub>.

Chas. A. ROUILLER.

β-Phenylcoumarins. I. G. BARGELLINI AND G. LEONARDI. Ist. chim. r. univ. Roma. Gass. chim. ital., 41, I, 737-46.—β-Phenyldaphnetin (Kostanecki and Weber, Ber., 26, 2907) is obtained by b. 2,3,4-(HO)<sub>2</sub>C<sub>2</sub>H<sub>2</sub>Bz with NaOAc and Ac<sub>2</sub>O and hydrolyzing the product with H<sub>2</sub>SO<sub>4</sub>. Similarly, from 2,4-dihydroxy-4'-methoxybenzophe-C(C<sub>2</sub>H<sub>4</sub>OMe): CH,

none is obtained 4-hydroxy-4'-methoxy-β-phenylcoumarin, HOC<sub>0</sub>H<sub>2</sub> O.CO

yellowish needles, m. 261-3°; sol. in alkalies with yellow color and green fluorescence; 4-acetate, woolly needles, m. 185-6°; 4-methyl ether, m. 156°. 2,4-Diacetoxy-4'-methoxy-bensophenone, needles, m. 128-30°, is also formed as a by-product. From 2,4,4'-tri-hydroxybenzophenone is obtained 4,4'-dihydroxy-β-phenylcoumarin, yellowish needles, m. 238-40°; 4,4'-diacetate, needles, m. 189-90°. 2,4,4'-Triacetoxybenzophenone, needles, m. 96-8°.

β-Phenylcoumarins. II. G. BARGELLINI AND G. FORLI-FORTI. Ist. chim. r. univ. Roma. Gass. chim. ital., 41, I, 747-56; cf. preceding abstr.—Cyanoacstoanisols, ρ-MeOC<sub>2</sub>H<sub>4</sub>COCH<sub>2</sub>CN, from the chloro compd. and KCN in H<sub>2</sub>O-EtOH, clusters of needles, m. 128-30°, condenses with m-C<sub>2</sub>H<sub>4</sub>(OH)<sub>3</sub> in the presence of ZnCl<sub>2</sub> and glacial AcOH or of 73% H<sub>2</sub>SO<sub>4</sub>, forming the 4-hydroxy-4'-methoxy-β-phenylcoumarin described in the preceding abstr. When H<sub>2</sub>SO<sub>4</sub> is used as the condensing agent a substance sol. in H<sub>2</sub>O and sepg. from alc. in needles m. 234-6° is also formed. With dil. KOH it evolves NH<sub>2</sub> and gives the above coumarin; it is probably the corresponding nitrile or amide. With 1,3,5-C<sub>2</sub>H<sub>4</sub>(OH)<sub>8</sub> is obtained in small amt. a compound in reddish yellow flocks, m. about 200°; acetate, needles, m. 179-80°. Chloroacetoveratole, (MeO)<sub>2</sub>C<sub>2</sub>H<sub>3</sub>COCH<sub>3</sub>Cl, from (HO)<sub>2</sub>C<sub>2</sub>H<sub>4</sub>COCH<sub>3</sub>Cl and Me<sub>2</sub>SO<sub>4</sub> or o-(MeO)<sub>3</sub>C<sub>4</sub>H<sub>4</sub> and CICOCH<sub>3</sub>Cl, in the presence of AlCl<sub>3</sub>, white scales, strongly attacks the skin and the

CICOCH<sub>2</sub>CI, in the presence of AlCl<sub>2</sub>, white scales, strongly attacks the skin and the mucous membranes. The yield is very small. Cyanoacstoveratrole, needles, m. 134-5°. With m-C<sub>2</sub>H<sub>4</sub>(OH)<sub>2</sub> and 73% H<sub>2</sub>SO<sub>4</sub> it gives a compound, m. 228-9°. 1,3,5-(HO)<sub>2</sub>C<sub>2</sub>H<sub>4</sub> forms a reddish yellow condensation product.

CHAS. A. ROUILLER.

Behavior of some Aliphatic Iodo Acids in the Organism. G. PONZIO. Ist. chim.

Behavior of some Aliphatic Iodo Acids in the Organism. G. Ponzio. Ist. chim. farm. tossic. r. univ. Sassari. Gass. chim. ital., 41, I, 781-7.—The following Ca salts of iodo acids are well tolerated by the organism and absorbed with elimination of I in the urin, while the corresponding amides pass unchanged through the gastro-enteric canal and are excreted with the feces. Calcium 2-iodopalmitate, [Me(CH<sub>2</sub>)<sub>12</sub>-CHICO<sub>2</sub>]<sub>2</sub>Ca, stable, amorphous powder, insol. in H<sub>2</sub>O or EtOH. The absorption becomes noticeable in 2 days and in 7-8 days no more I is found in the urin. Amide, stable laminas, m. 108°. It is obtained either from the acid through the chloride or from 2-bromopalmitamide, laminas, m. 85°, by b. with 5% KI in 96% alc. 2-lodo-

stearic acid, prepared like the iodopalmitic acid (Gasz. chim. ital., 35, II, 132), laminas, m. 66°, stable in the light. Calcium salt. Amide, laminas, m. 112°. 2-Bromostearamide, laminas, m. 91°. Chas. A. Rouller.

Transformation of Nitro Aldehydes into Cyano Aldehydes. G. Ponzio. Ist. chim. farm. tossic. r. univ. Sassari. Gazz. chim. ital., 41, I, 787-93.—o-O<sub>2</sub>NC<sub>6</sub>H<sub>6</sub>NHN: CPhCN (cf. C. A., 5, 694) is obtained exclusively when the corresponding nitro compd. is b. in alc. with KCN. Similarly is obtained phenylcyanoformaldehyde-m-nitrophenyl-hydrazone, yellow prisms, m. 197-8°, sol. in alkalies with yellow color, from phenyl-nitroformaldehyde-m-nitrophenylhydrazone, brick-red prisms, m. 132° (decomp.). Phenylnitroformaldehyde-o,p-dinitrophenylhydrazone, yellowish brown, flattened needles, m. 152° (decomp.). Chas. A. Rouiller.

Action of Nitrous Acid on Substituted Hydrazidines. G. Ponzio and C. Gastaldi. Ist. chim. farm. tossic. r. univ. Sassari. Gazz. chim. ital., 41, 1, 793-6.—When p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NHN: CPhNH<sub>2</sub> is treated with KNO<sub>2</sub> in HCl or with isoamyl nitrite in glacial AcOH, it gives benzenyl-p-nitrophenylnitrosohydrazidine, O<sub>4</sub>NC<sub>6</sub>H<sub>4</sub>N(NO)N: CPhNH<sub>2</sub>, yellowish laminas, m. 127° (decomp.), which, when b. with H<sub>2</sub>O<sub>4</sub> yields a mixt. of O<sub>4</sub>NC<sub>6</sub>H<sub>4</sub>NHN: CPhNH<sub>2</sub> and O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NHNHBz. Chas. A. ROULLER.

Mechanism of the Opening of the Picenic Ring in Pinene Derivatives. GUIDO CUSMANO. Gazz. chim. ital., 41, II, 1-8; cf. C. A., 5, 276. C. A. R.

Tetrahydroxybenzenes. G. Bargeillini and Leda Bini. Ist. chim. r. univ. Roma. Gazz. chim. ital., 41, II, 8-20; cf. C. A., 5, 1279. C. A. R.

Triazole and its Derivatives. Guido Pellizzari. Ist. chim. gen. r. univ. Genova. Gazz. chim. ital., 41, II, 20-42.—A brief summary of P.'s earlier publications on triazole and its derivs. is given. The following has not yet been published: C-Phenyltriazole, C-dimethyltriazole, C-diphenyltriazole, I-phenyl-3-methyl-1,2,4-triazole, I-phenyl-5-methyl-1,2,4-triazole (identical with Bladin's "I-phenyl-3-methyltriazole" (Ber., 18, 1544), and triphenyl-1,2,4-triazole were obtained by P.'s method (treatment of an acyl hydrazide, RCONHNH<sub>2</sub>, with an amide, R'CONH<sub>2</sub>). I-Phenyl-1,2,4-triazole nitrate, needles, m. 141°; picrate, m. 159°. I-Phenyl-3,5-dimethyl-1,2,3-triazole, m. 43°, b. 281°; chloroplatinate, orange, prismatic needles with 2 H<sub>2</sub>O, m. 185-6° and (anhydrous) 195°; picrate, yellow needles, m. 156°. I-Phenyl-2-methyl-1,3,4-triazole, lamellas with I H<sub>2</sub>O, m. 68°, (anhydrous) m. 112°; picrate, m. 134°; chloroplatinate, orange laminas, m. 206°; tetrachloroplatinate, light yellow amorphous ppt. Chas. A. ROUILLER.

1-Phenyl-5-methyl-1,2,4-triazole and Cyanophenylacetamidine. Guido Pelliz-Zari. Ist. chim. gen. r. univ. Genova. Gazz. chim. ital., 41, II, 93-100; cf. preceding abstr.—1-Phenyl-5-methyl-1,2,4-triazole, prepared by Bladin's method (Ber., 18, 1544), b. 273°. If, instead of distilling the carboxylic acid, as in Bladin's method, it is heated at 170° for 1 hr., there is formed, besides the triazole, the compd. m. 193° supposed by Bamberger and Gruyter (Ber., 26, 2385) to be the triazole but which is really cyanophenylacetamidine, PhN: CMeNHCN sol. in cold alkalies, and decomp. by hot alks. into AcNHPh and NCNH2. In conc. HCl it forms carboxamidophenylacetamidine, PhN: CMeNHCONH3, laminas, m. 180° (decomp.) when heated fast, 166°, when heated slowly (20-5 min.). The PhN: CMeNHCN was also synthesized from NCNH3 and PhN: CMeOEt.

Hydroxyhydroquinone Derivatives. G. BARGELLINI. Ist. chim. r. univ. Roma. Gazz. chim. ital., 41, II, 43-8; cf. C. A., 5, 2098. C. A. R.

Strychnine and Brucine. II. R. CIUSA AND SCAGLIARINI. Ist. chim. univ. Bologna. Gazz. chim. ital., 41, II, 48-52; cf. C. A., 5, 1279. C. A. R.

Oxidation of Phthalacene. D. MAROTTA. Ist. chim. gen. r. univ. Palmero. Gazz. chim. ital., 41, II, 59-63.—Phthalacene, heated 5 hrs. at 200° with 20 pts. HNO<sub>8</sub> (d. 1.035) and then 5 hrs. longer with 10 pts. more of acid, gives 3,4,5,6-di-o-benzoylene-bensoic acid,

reddish brown crystals, m. 299-300°. Potassium salt, yellow powder, easily hydrolyzed. Ethyl ester, from the Ag salt and EtI, yellow, silky needles, m. 230°. HI and P reduce the acid to 3,4,5,6-di-o-bensylenebensoic acid, slightly yellow needles, m. above 300°. Alkalies in large excess ppt. the easily hydrolyzed alkali salts as yellow powders. Ethyl ester, brown crystals, m. 136-7°. Chas. A. Rouiller.

Action of Acetyl Chloride on Acetylbiuret. A. Ostrogovich. Ist. chim. univ. Bucarest. Gasz. chim. ital., 41, II, 70-4.—Biuret, heated 2-3 hrs. with excess of NH.CMe: N

AcCl at 100° and then 4 hrs. at 140-5°, gives methyldioxytriazine, | | . CO.NH — CO CHAS. A. ROUILLER.

General Addition Reaction of Aralkylideneurethans to β-Dicarboxylic Compounds. G. BIANCHI AND R. SCHIFF. Lab. chim. farm. r. univ. Pisa. Gazz. chim. ital., 41, II, 81-93.—Ethyl bensylideneurethanacetoacetate, PhCH(NHCO<sub>2</sub>Et)CHAcCO<sub>2</sub>Et, from AcCH<sub>2</sub>CO<sub>2</sub>Et, H<sub>2</sub>NCO<sub>2</sub>Et, BzH and a few drops of conc. HCl in 95% alc., micro-cryst. powder, m. 96-7°, very stable towards mineral acids. PhCH(NHCO<sub>2</sub>Et)<sub>2</sub> is first formed in the reaction. Ethyl benzylideneurethanbenzoylacetate, m. 97. Benzylideneurethanacetylacetone, m. 101°. Ethyl cinnamylideneurethanacetoacetate, m. 92-3°. Cinnamylideneurethanacetylacetone, m. 102°. Anisylideneurethanacetylacetone, m. 98°. Salicylideneurethanacetylacetone, m. 128-30°.

Action of Cyanoacetic Ester on o- and p-Hydroxybenzaldehydes in the Presence of Ammonia. Mario Sclavi. Atti accad. sci. Torino, 46, 181-94; through J. Chem. Soc., 100, I, 398.—From 1 mol. p-HOC<sub>2</sub>H<sub>4</sub>CHO, 2 mols. NCCH<sub>2</sub>CO<sub>2</sub>Et and 3 mols. NH<sub>2</sub> at room temp. are obtained 3 products: (1) NH<sub>4</sub> salt of 2,6-diketo-3,5-dicyano-4-p-hydroxyphenylletrahydropyridine, lustrous white crystals; ammonium salt,

does not m. below 300° nor lose NH<sub>2</sub> even at 100°; the ferric, ferrous, copper, potassium, magnesium (with 6 H<sub>2</sub>O), barium (with 6 H<sub>2</sub>O) and silver (with 1 H<sub>2</sub>O) salts are cryst. (2)  $\alpha$ -Cyano- $\beta$ -hydroxyphenylacrylamide, yellow crystals, m. 245° (loss of NH<sub>2</sub>). (3)  $\alpha$ -Cyano- $\beta$ -hydroxyphenylpropionamide, m. 156°. With the o-HOC<sub>2</sub>H<sub>4</sub>CHO the chief products are EtO<sub>2</sub>CC(CN): C(C<sub>2</sub>H<sub>4</sub>OH)CH(CN)CO<sub>2</sub>Et and an ammonium salt which was not identified. Chas. A. ROUILLER.

Crystallographic Study of some Nitrophenylmethylacrylic Derivatives. Francesco Ranfaldi. Ist. min. r. univ. Napoli. Rend. accad. sci. fis. mat. Napoli, [3a] 16, 225-34.—o-Nitrophenylmethylacrylic acid,  $O_2NC_4H_4CH$ : CMeCO<sub>2</sub>H, m. 198°, monoclinic prisms, a:b:c=1.3446:1:1.4562,  $\beta$  92° 24′ 51″, (100), (001), (101), (102), (110), (122), (122), the form (100) always predominating, quite easy and perfect cleavage along (101), plane of the optical axes (010), acute bisectrix normal to (101). m-Nitrophenylmethylacrylic acid, m. 203.5°, acicular crystals, could not be obtained in measurable form. p-Nitrophenylmethylacrylic acid, m. 208°, triclinic,

pinacoidal crystals, a:b:c=1.2867:1:1.4602,  $\alpha 84^{\circ} 42'8''$ ,  $\beta 83^{\circ} 31'31''$ ,  $787^{\circ} 35'18''$ , (100), (001), (101), (101), (110), (110), (112), (114), (112), (114), the pinacoid (110) predominating. Sodium o-nitrophenylmethylacrylate, rhombic, bisphenoidal crystals, a:b:c=1.3940:1:2.0544, (100), (001), (304), (110), x(122), the pinacoid (100) predominating, plane of the optical axes parallel to (010), acute, positive bisectrix normal to (100). Thus there is no crystallographic resemblance between the o- and p-acids, but the morphotropic relations between the o-acid and its Na salt are marked.

Velocity of Addition of Bromine to the Imides of some Substituted Maleinamic Acids. II. A. PIUTTI AND G. CALCAGNI. Ist. chim. farm. r. univ. Napoli. Rend. accad. sci. fis. mat. Napoli, [3a] 16, 255-61; cf. Ibid., 15, 18.—Detns. of the velocity of addition of Br to the white and yellow methoxy- and ethoxyphenylmaleinimides and the white hydroxyphenylmaleinimide showed that the white compds. add Br. more rapidly (reaction complete in about 50 ds.) than the yellow compds. (75 ds.), confirming the structures (I) and (II) previously assigned to the white and yellow

$$\begin{array}{c|c} \text{HC.CO} & \text{HC.C}(: \text{NC}_{\delta}\text{H}_{\delta}\text{OR}) \\ \text{HC.CO} & \text{HC.CO} & \text{(II)} \\ \end{array}$$

compds., resp. (cf. C. A., 5, 1403).

CHAS. A. ROUILLER.

AZONY Compounds. A. ANGELI AND LUIGI ALESSANDRI. R. ist. studi sup. Atti accad. Lincei, 20, II, 170-6; cf. C. A., 5, 3405.-p-Nitroasobenzene, from p-O<sub>2</sub>NC<sub>2</sub>H<sub>4</sub>NH<sub>2</sub> and PhNO in cold glacial AcOH, orange-red laminas from alc., m. 135°; dissolves easily in HNO<sub>2</sub> (d. 1.45), but p,p-dinitroaxobenzene, m. 221°, soon deposits as a red powder. H<sub>2</sub>O<sub>2</sub> in cold glacial AcOH slowly oxidizes the PhN<sub>2</sub>C<sub>2</sub>H<sub>4</sub>NO<sub>2</sub> to p-nitroazoxybenzene, intensely yellow crystals, m. 149°; it forms large crystals or slender, transparent needles, each distinct from the other, while the compd. obtained by Zinin (Ber., 32, 3256) from HNO, and Ph.N.O m. 153° and forms opaque, microscopic needles in leaf-like or feathery clusters of a light yellow color with a tendency to green. In the light Z.'s compd. is stable while the authors' turns orange and finally forms a minium-red powder, the PhN<sub>2</sub>C<sub>2</sub>H<sub>4</sub>NO<sub>2</sub> being apparently regenerated. At room temp. HNO, (d. 1.48) does not act on Z.'s compd. in a short time, but on prolonged treatment it gives the same (p-O<sub>2</sub>NC<sub>2</sub>H<sub>4</sub>)<sub>2</sub>N<sub>3</sub> formed by the authors' product, while very conc. acid (d. 1.52) converts it into m-trinitroazoxybenzene. If mixed with a little I and then treated with Br, Z.'s compd. is unaltered while the other gives a bromo derivative, m. 199°. It is possible that the Zinin compd. has the formula PhN: N-(: O)C<sub>2</sub>H<sub>4</sub>NO<sub>2</sub> and the authors' the formula PhN(: O): NC<sub>2</sub>H<sub>4</sub>NO<sub>2</sub>.

Some Derivatives of Hydroxyhydroquinone. VI. G. BARGELLINI AND ERMANNO MARTEGIANI. Ist. chim. r. univ. Roma. Atti. ascad. Lincei, 20, II, 183-90; cf. C. A., 5, 3683.—1,2,4-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>2</sub> and BzCl condense in CS<sub>2</sub> in the presence of AlCl<sub>2</sub>, giving 2,4,5-trimethoxybenzophenone, yellowish needles, m. 97°; phenylhydrazone, scales, m. 178-9°, sol. in conc. H<sub>2</sub>SO<sub>4</sub> with greenish yellow color; and 2-hydroxy-4,5-dimethoxybenzophenone, flattened, greenish yellow prisms, m. 106-7°, gives an orange-yellow color with conc. H<sub>2</sub>SO<sub>4</sub> and a yellowish green color with alc. FeCl<sub>2</sub>; acetate, yellowish needles, m. 108-10°. HBr (d. 1.47) in glacial AcOH hydrolyzes both the tri- and dimethoxy compds. to 2,4-dihydroxy-5-methoxybenzophenone, yellow needles, m. 183-5°. From p-MeOC<sub>2</sub>H<sub>4</sub>COCl are obtained 2,4,5,4'-tetramethoxybenzophenone, yellowish cryst. powder, m. 122-4°; phenylkydrazone, m. 173-4°; and 4,5,4'-trimethoxy-2-hydroxybenzophenone, yellow needles, m. 127-8°. PhCH<sub>2</sub>CO<sub>2</sub>H gives 2,4,5-trimethoxydesoxybenzoin, leaves, m. 276-7°; phenylkydrazone, yellowish cryst. powder, m. 142-3°; and 4,5-dimethoxy-2-hydroxydesoxybenzoin, leaves, m. 276-7°; phenylkydrazone, yellowish cryst. powder, m. 142-3°; and 4,5-dimethoxy-2-hydroxydesoxybenzoin, leaves, m. 04°. Chas. A. ROUILLER.

Strychnins and Brucine. R. CIUSA AND G. SCAGLIARINI. Ist. chim. gen. univ. Bologna. Atti accad. Lincsi, 20, II, 210-6; cf. C. A., 4, 2457; 3, 1279.—Löbisch and Schoop's "β-bromostrychnine" (Monatsh., 6, 585) is really identical with the bromostrychnine m. 222-3°, and does not lose HBr when heated in sealed tubes with alc. KOH. With Br in glacial AcOH, brucine gives a compound which has probably the comp. C<sub>m</sub>H<sub>m</sub>O<sub>4</sub>N<sub>2</sub>Br.HBr.H<sub>2</sub>O, yellowish needles, does not m. below 270°; chloroplatinats, (C<sub>m</sub>H<sub>m</sub>O<sub>4</sub>N<sub>2</sub>Br)<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>.H<sub>2</sub>O, red, cryst. powder, decomp. without m. In cold HCl, strychnine gives with KClO<sub>2</sub> Minuni's tetrachlorostrychnine (Gazz. chim. ital., 30, I), but if the soln. is not kept cold, a compd. which is apparently octochlorostrychnine is formed; a yellowish cryst. powder, sol. in alkalies with dark red color, insol. in carbonates, blackens without m.; p-nitrophenylhydrazone, orange powder. The mono- and dibromostrychnine derivs. described in this and the earlier article are as toxic as strychnine itself, while the chloro compds. are not poisonous. C. A. R.

Influence of Auxochromes on Phototrophy. M. PADOA AND L. SANTI. Lab. chim, gen, r. univ. Bologna. Atti accad, Lincei, 20, II, 196-200,-In general, the phototropy of hydrazones of aldehydes containing auxochromes is more marked than that of derivs. of aldehydes containing no such groups. An auxochrome in the pposition seems to have more influence than one in the o-position. A comparative study of compds. in which the auxochrome is in the hydrazine group shows some irregularities. From p-MeOC<sub>2</sub>H<sub>4</sub>NHNH<sub>2</sub>, which is easily prepared by diazotizing MeOC.H.NH. and reducing the product with SnCl., were obtained the following compds.: Benzaldehydeanishydrazone, faintly yellow, silky needles, m. 123°, phototropic. Anisalanishydrazone, yellow scales, m. 126°, non-phototropic. Cinnamalanishydrazone, short opaque needles, intensely yellow, grouped in rosets, m. 126.5°, phototropic. Cuminalanishydrazone, light yellow needles, m. 99°, phototropic. Piperonalanishydrasone, greenish yellow needles, m. 134-5°, phototropic. p-Tolualanishydrasone, vellow scales, m. 131°, non-phototropic. Vanillin anishydrazone, minute, pale vellow prisms, m. 125-6°, photetropic. Salicylalanishydrazone, greenish yellow needles, m. 132°, non-phototropic. CHAS. A. ROUILLER.

Simultaneous Reduction and Oxidation Catalysis. N. Zelinskii and N. Glinka. Lab. org. and analy. Chem., Univ. Moscow. Ber., 44, 2305-11.—At the ordinary temp., Pd black and H convert Me  $\Delta'$ -tetrahydroterephthalate into a mixture of Me hexahydroterephthalate and Me terephthalate; this latter crystallizes from the Et<sub>2</sub>O. The esters are formed in the ratio 2 or 3: 1 of the phthalate. The Pd black is best prepared by mixing 1 l. of aq. PdCl<sub>2</sub>.2NH<sub>2</sub> (2%) with HCO<sub>2</sub>H (d. 1.2) and adding to the warm liquid 50 cc. of aq. KOH (20%). The Pd black glows in air when it is moistened with alc. and it decomp. HCO<sub>2</sub>H quickly, even at the ordinary temps., into CO<sub>2</sub> + H. I. BISHOP TINGLE.

 $\Delta^{\nu a}$ -Dihydrobenzene. N. Zelinskii and A. Gorskii. Chem. Lab. org. and analy. Chem., Univ. Moscow. Ber., 44, 2312-6.—In reply to Brühl (C. A., 3, 323), who criticized the authors' earlier work (C. A., 2, 2944), it is pointed out that, up to the present time, no single instance of exaltation has been observed among the simple non-substituted cyclodienes. It reply to Harries and von Splawa-Neyman (C. A., 3, 1174) it is shown that the presence of even 48.48% of 1,3-dimethylcyclohexene is insufficient to mask the exaltation of 1,3-dimethylcyclo-1,3-diene. There is no basis for the doubts expressed by Harries and von Splawa-Neyman regarding the purity of the authors' cyclohexadiene. 1-Bromocyclo-1-hexene formula (I) below, is formed

together with cyclo-1,3-hexadiene, from 1,2-dibromocyclohexane and quinoline. Yield, 15-20% when the dibromo compd. is dropped into b. quinoline. The use of quinoline (3-4 parts) and dibromo compd. (1 part) favors the production of the hexadiene, whereas with more quinoline the yield of (I) in increased. The physical constants of (I) are as follows: b.  $164-6^{\circ}$ ,  $b_{20}$  69°,  $d_4^{20}$  1.3901,  $n_D^{20}$  1.5134, mol. ref. 34.84. It is oxidized readily by "permanganate" to adipic acid. Dibromo compound, CaHaBra, from (I) and Br, in CHCla; liquid, bis 138-40°. When distilled with quinoline (I) is not changed. Crossley's hydrobromide (J. Chem. Soc., 85, 1422 (1904)) is prepared from cyclo-1,3-hexadiene and HBr, in well cooled AcOH; b<sub>20</sub> 80-0.5°. Yield, 80%. It gives with Br a tribromo compound, C.H.Br.; b., 150°. The hydrobromide cannot have formula (II) because, when oxidized with "permanganate", it gives an acid, CaH10Os; crystals from CHCl2 + ligroin, m. 93-4°; b20 240° (slight decomp.). It is not changed by warming with conc. H<sub>2</sub>SO<sub>4</sub> and does not form an anhydride when b. Silver salt, CaHaOaAga. It is very probable that the parent hydrocarbon is not cyclo-1,3-hexadiene. J. BISHOP TINGLE.

Diphenylstibine Compounds. A. MICHAELIS AND ARWED GÜNTHER. Chem. Inst., Univ. Rostock. Ber., 44, 2316–20.—Diphenylchlorostibine, Ph<sub>2</sub>SbCl, is prepared by heating triphenylstibine, SbCl<sub>2</sub> and xylene at 240°, during 48 hrs., the products being separated by distillation and the stibine purified by warming with dil. HCl; white crystals, m. 68°, decomp. when rapidly heated. It is stable towards H<sub>2</sub>O and it attacks the skin. Aq. Na<sub>2</sub>CO<sub>2</sub> converts the chloride into diphenylstibine oxide, (Ph<sub>2</sub>Sb)<sub>2</sub>O; white, needle-shaped crystals from alc., m. 78°. Diphenylstibine sulfide, (Ph<sub>2</sub>Sb)<sub>2</sub>S, is obtained from the oxide and H<sub>2</sub>S, in alc.; long, white needles from alc., m. 69°. Dry Cl and diphenylchlorostibine give diphenylstibine trichloride, Ph<sub>2</sub>SbCl<sub>2</sub>.H<sub>2</sub>O, m. 176° not 180°. Hot, dil., aq. NaOH hydrolyzes it to diphenylstibinic acid, Ph<sub>2</sub>SbOOH.

Organometallic Ester Compounds. I. Iodostannic Ester Compounds. Bruno EMMERT AND WILHELM ELLER. Chem. Inst., Univ. Würzburg. Ber., 44, 2328–31.—Diethyl diidostannicdiacetate, I<sub>2</sub>Sn(CH<sub>2</sub>CO<sub>2</sub>Et)<sub>2</sub>, is prepared from Et iodoacetate, Sn foil and a trace of I, at 60–70°, during 6–10 hrs., moisture being excluded; colorless, odorless needles from CCl<sub>4</sub> + abs. Et<sub>2</sub>O, m. 101.5° (cor.). Yield, 84%. It is hydrolyzed by H<sub>2</sub>O, or similar agents, preferably Ba(OH)<sub>2</sub>, to AcOEt, HI and SnO<sub>2</sub>. With PhMgBr, or EtMgBr it gives SnPh<sub>4</sub> and SnEt<sub>4</sub>, resp. Diethyl diiodostannicdi-obensoate, I<sub>2</sub>Sn(C<sub>2</sub>H<sub>4</sub>CO<sub>2</sub>Et)<sub>2</sub>, is prepared from Et o-iodobenzoate, in a similar manner to the acetate, at 150°, during 5 days; pale grayish yellow, microscopic needles from abs. Et<sub>2</sub>O, decomp. without m. above 300°.

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Codeine Oxide. Martin Freund and Edmund Speyer. Chem. Inst. Phys. Assoc. Acad., Frankfurt a/M. Ber., 44, 2339-53; cf. C. A., 5, 702.—Codeine-oxide-sulfonic acid,  $C_{18}H_{20}O_{3}(: N: O)SO_{2}H$ , is prepared by adding gradually codeine oxide (50 g.) to a cool mixture of Ac<sub>2</sub>O (250 cc.) and conc.  $H_{2}SO_{4}$  (25 cc.); the product is warmed with alc. and the crystals purified by soln. in dil., aq. NaOH and pptn. with acid. Small, hexagonal crystals, decomp. 272°; in 2 N aq. KOH,  $\alpha_{20}^{20}$ —115.4°. It reduces hot Fehling's soln. Yield, 100% of the oxide. Polassium salt, cryst. At 180°, during 4 hrs.,  $H_{2}O$  hydrolyzes the sulfonic acid to codeine, which was isolated as the hydriodide. Codeinesulfonic acid,  $N: C_{18}H_{20}O_{2}SO_{2}H$ , is prepared by warming the oxide sulfonic acid with aq.  $SO_{2}$ ; small, well-developed, prismatic crystals and silky needles which are mutually transformable from  $H_{2}O$ , decomp. above 300°. It regenerates the oxide-sulfonic acid by the action of  $H_{2}O_{3}$ , in aq.  $NH_{2}$  and is hydrolyzed by  $H_{2}O$ , under pressure, to codeine. Codeinesulfonic acid is also formed, in better yield, from the oxide-sulfonic acid, aq. NaOH and Na hyposulfite; in 2 N aq. KOH,  $\alpha_{20}^{20}$ —136.3°. It reduces hot Fehling's soln. slightly. With cold HNO<sub>3</sub> (d. 1.3)

codeinesulfonic acid is converted into nitrocodeine (m. 217°). Methylkydroxycodeinesulfonic acid, O: C14Ha(OMe)(OH)a(C2H4NMe2)SO2H, is obtained from codeinesulfonic acid, alc., aq. KOH and MeI; needles from alc. +  $H_2O$ , decomp. 284°; in  $H_2O$ ,  $\alpha_{20}^{20}$ -63.2°. When b. with aq. KOH it is hydrolyzed to tetramethylethylenediamine, C<sub>2</sub>H<sub>4</sub>(NMe<sub>2</sub>)<sub>2</sub>, and a compound, C<sub>12</sub>H<sub>11</sub>O<sub>2</sub>SK; well-developed plates from H<sub>2</sub>O<sub>2</sub> decomp. 295°. It decolorizes aq. KMnO4 and is not decomp. by HCl. A second compound, together with tetramethylethylenediamine, is formed by heating methylhydroxycodeinesulfonic acid directly with conc. aq. KOH; colorless, unstable plates from H<sub>2</sub>O. Cold, conc. H<sub>2</sub>SO<sub>4</sub> converts codeine-oxide-sulfonic acid into  $\beta$ -codeinesulfonic acid, C12H12O2NS, or C12H21O2NS, which is purified by soln. in dil., aq. NH2 and pptn. with H<sub>2</sub>SO<sub>4</sub>; bundles of long, microscopic plates, decomp. and chars about 243°; in highly dil. aq. KOH,  $\alpha_D^{20}$  —190.1°. It does not reduce Fehling's soln. Digestion with aq. SO, transforms it into an isomeric \( \gamma\)-codeinesulfonic acid, interlaced needles from alc., decomp. about 280°. It does not reduce Fehling's soln. Codeine-oxidesulfonic acid hydrate, C18H202NS, is prepared by heating codeine-oxide-sulfonic acid with aq. K<sub>2</sub>CrO<sub>4</sub> (10%), HCHO being evolved; small needles from H<sub>2</sub>O. It is sol. in dil., aq. NH, and is repptd. by acids. Yield, about 40% of the parent acid. At 180°, during 8 hrs., it is hydrolyzed to a base, C<sub>17</sub>H<sub>18</sub>O<sub>2</sub>N, which is isomeric with morphine; opaque, prismatic plates, or long needles, m. 180°. It gives a violet color with aq. HCHO + H.SO. Hydrochloride, C1, H1, O2N.HCl, well developed needles from H2O, decomp. about 310°. Codeinesulfonic acid and well cooled, conc. H.SO. form a compound, C12H12O2NSO2H; hair-like needles from aq. NH2 + dil. HCl, decomp. 285-90°. When a mixture of codeine oxide, Ac<sub>2</sub>O and conc. H<sub>2</sub>SO<sub>4</sub>, as described above, is allowed

to become hot  $\alpha$ -codeine-oxide-sulfonic acid,  $C_{18}H_{20}O_{2}$   $O_{18}O_{$ 

deposited from H<sub>2</sub>O in opaque prisms containing H<sub>2</sub>O, becomes anhydrous 130°. It reduces warm Fehling's soln. and is transformed into codeine-oxide-sulfonic acid by gently heating with aq. NaOH (10%), dil. H,SO,, or dil. HCl. Yield, 70% of the codeine oxide. a-Codeine-oxide-sulfonic acid is converted by SO, into codeinesulfonic acid and is hydrolyzed to codeine by H<sub>2</sub>O, at 160°. With HNO<sub>2</sub> (d. 1.4) it gives nitro-a-codeine-oxide-sulfonic acid, O2NC10H10O2( : NO)SO2H; yellow plates from H2O, decomp. 167-70°. Warm. aq. SO, reduces it to α-nitrocodeine, C<sub>12</sub>H<sub>20</sub>O<sub>2</sub>NNO<sub>2</sub>; quadratic plates from alc., m. 197°. It is isomeric with Anderson's nitrocodeine (m. 217°). In hot H<sub>2</sub>O, Br-H<sub>2</sub>O converts α-codeine-oxide-sulfonic acid into a bromo compound, C<sub>1</sub>,H<sub>1</sub>,O<sub>2</sub>Br<sub>2</sub>; long needles, or rods from alc. NaOH + HCl, m. 258-60° (decomp.). Codeinesulfonic acid is devoid of physiological activity. Aminocodeine, C12H202NNH2, is prepared from either of the above nitrocodeines, by electrolytic reduction, in presence of dil. H<sub>2</sub>SO<sub>4</sub>, or by the action of Zn + dil. HCl; plates from alc., or CHCl<sub>2</sub> + Et<sub>2</sub>O, m. 228°. Hydrocklorids, C18H2O6NNH2.HCl.2H2O, needles by dissolving the base in HCl and adding aq. NH, in excess; decomp. 290°. Hydroxycodeine, C18HnO4N, is prepared by diazotizing the preceding compd.; almost colorless plates with I H<sub>2</sub>O from MeOH, becomes anhydrous 176-85°, m. 234°. It is isomeric with Knorr's 9or 10-hydroxycodeine. Hydrochloride, C<sub>18</sub>H<sub>21</sub>O<sub>4</sub>N.HCl.2H<sub>2</sub>O, colorless needles from J. BISHOP TINGLE. H<sub>0</sub>O.

Cotarnine. VI. MARTIN FREUND AND KARL LEDERER. Chem. Inst. Phys. Assoc. Acad., Frankfort a/M. Ber., 44, 2353-6; cf. Ibid., 39, 2229 (1906).—α-Phenylhydrotarconine formula (I) below, is prepared from tarconine methiodide and PhMgBr; bundles of white crystals from alc., softens 97°, m. 102°. It darkens in air. α-Butylhydrocotarnine (1-butyl-2-methyl-6,7-methylenedioxy-8-methoxytetrahydroisoquinoline) (II), is prepared in a similar manner to the Ph derivative (loc. cit.); oil. Sulfate and

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chloroaurate, oily. Hydrochloride, plates from alc., m. 215-6°. Hydrobromide, plates, m. 207-8°. Hydrodide, rods from alc., m. 190-1°. Picrate, plates from alc., m.

165-6°. Chloroplatinate, plates from alc., m. 205-6°. Methiodide, long, rhombic plates from alc., m. 190-3°.

J. BISHOP TINGLE.

Action of Organomagnesium Compounds on Hydrastinine. MARTIN FREUND AND KARL LEDERER. Chem. Inst. Phys. Assoc. Acad., Frankfort a/M. Ber., 44, 2356-62.— $\alpha$ -Methylhydrohydrastinine formula (I) below (R = Me), is prepared by

adding hydrastinine to excess of MeMgI, in abs. Et.O; yellow oil. Hydriodide, plates from H<sub>2</sub>O, or alc., m. 227°. Hydrochloride and sulfate, readily sol. Bromide (? hydrobromide), crystallizes readily. Methiodide, plates from H<sub>2</sub>O, m. 222-30°. α-Ethylhydrohydrastinine (I; R - Et), from EtMgBr; hard, white plates from ligroin, m. 70-1°. α-Phenylhydrohydrastinine (I, R = Ph), long crystals with 1 H<sub>2</sub>O from dil. alc., m. 88°. When anhydrous it is yellow and vitreous. a-Propylhydrohydrastinine (I; R = Pra), and the analogous compds. described below were prepared from hydrastinine hydrochloride and Grignard's reagent; oil. Hydriodide, plates from alc., m. 185-6°. Chloroplatinate, rhombic crystals from alc. + dil. HCl, decomp. 230°. Methiodide, plates from alc. + Et,O, softens 163°, m. 168-9°. α-Isopropylhydrohydrastinine (I;  $R = Pr^{\beta}$ ), oil. Hydrobromide, needles from alc., m. 190°. Hydriodide, white rhombic plates from alc. + SO<sub>2</sub>, m. 217-8°. Picrate, plates from MeOH, m. 143-5°. Chloroplatinate, needles from alc. + HCl, m. 223°. Methiodide, bundles of needles from alc., m. 219-20°. a-Butylhydrohydrastinine (I; R = Bu), oil. Picrate, plates from alc., m. 147-8°. Chloroplatinate, long, rhombic plates from alc. + HCl, m. 222-3°. Methiodide, plates from abs. alc., m. 205-6°. α-Isobutylkydrohydrastinine (I; R = Me<sub>2</sub>CHCH<sub>2</sub>), oil. Picrate, prisms from MeOH + Et<sub>2</sub>O, softens 125°, m. 130°. Chloroplatinate, long, quadratic plates from alc. + dil. HCl, decomp. 220°. Methiodide, bundles of plates from abs. alc., m. 197-8°. α-Benzylhydrohydrastinine (I; R - PhCH<sub>2</sub>), from PhCH<sub>2</sub>MgCl; oil. Hydrochloride, hexagonal crystals from aq. HCl, from alc. crystals with 1 EtOH, m. 182°, previously softening. Hydrobromide, crystals with 1 H<sub>2</sub>O from dil. alc., m. when anhydrous 187-8°. Hydriodide, hexagonal plates from alc., m. 195-6°. Acid sulfate, crystals with 1 H<sub>2</sub>O from dil. alc., when anhydrous softens 185°, m. 189°. Picrate, octahedral crystals from MeOH, m. 178-80°. Chloroplatinate, plates from alc. + HCl, decomp. 224°. Methiodide, small rods from abs. alc., m. 245°.  $\alpha, p$ -Anisylhydrohydrastinine (I; R = p-MeOC<sub>2</sub>H<sub>2</sub>), from p-MeOC. H.MgI; bundles of long rods from abs. alc., m. 98-9°. Hydrochloride, prepared from dry HCl, in Et<sub>2</sub>O; needles from alc. Hydrobromide, needles from alc., m. 243-4°. Hydriodide, needles from alc., m. 223-4°. Methiodide, prisms from m. 213-4°.  $\alpha,\alpha'$ -Naphthylhydrohydrastinine (I;  $R = \alpha - C_{10}H_7$ ),

 $\alpha$ -C<sub>10</sub>H<sub>2</sub>MgBr; rhombic plates from abs. alc., softens 125°, m. 127-8°. Hydrockloride, crystals from alc., m. 254-5°. Hydrobromide, needles from dil. alc., m. 265°. Hydrobromide, needles from dil. alc., m. 265°. Acid sulfate, interlaced crystals from alc. + Et<sub>2</sub>O, softens 225°, decomp. 228-9°. Picrate, plates from alc., decomp. 201°. The yield of the above alkyl and aryl  $\alpha$ -hydrastinine compds. is 50%, half the hydrastinine employed being recovered. From hydrastinine hydrochloride the yield is quant.

J. BISHOP TINGLE.

Occurrence of Quinoidal Properties in Anthraquinone Derivatives. R. Scholl. Ber., 44, 2370-7; cf. C. A., 5, 3249.—Benzo-1-nitro-1,2-benzanthraquinone formula (I) below, is prepared from 1,2-benzanthraquinone, HNO<sub>4</sub> (d. 1.52), glacial AcOH, Ac<sub>4</sub>O

and a little carbamide, the product is treated with CHCla, in which the following compd. is the more readily sol.; yellow plates or prisms from C<sub>e</sub>H<sub>a</sub>, m. 277-8°. Benzo-4-nitro-1,2-benzanthraquinone (II), is formed together with (I); needles from C.H., m. 250-1°. B., aq. Na,S, or PhN,H, reduces it to bens-4-amino-1,2-bensanthraquinone; red crystals from toluene, or PhNO<sub>2</sub>, m. 215°. In conc. H<sub>2</sub>SO<sub>4</sub> the color is reddish brown. Its vat dye, prepared by the action of alkaline "hyposulfite" is yellowish red and shows no affinity for unmordanted vegetable fibers. It fails to give a vat dye by b. with SbCl<sub>a</sub> and PhNO<sub>2</sub>, or by the action of KOH with, or without alc., at 300°. O,N-Dihydro-2,o-(N)-indolognthrone (III), is formed by b. (I) with PhN,H, during 5 mins. and is purified by soln. in dil., aq. NaOH, containing a little Na hyposulfite; green, amorphous powder. Its solns. are brown to brownish red with a green fluorescence. In conc. H<sub>2</sub>SO<sub>4</sub> the color is reddish brown. In aq. NaOH, air oxidizes it to 2.9-(N) indoloanthrone (IV); violet-brown, amorphous flocks, softens about 160°, not m. 360°. With PhN<sub>2</sub>H<sub>2</sub> it regenerates (III). In conc. H<sub>2</sub>SO<sub>4</sub> the color is reddish brown. The above formulas appear to be essentially hypothetical. They are discussed at considerable length. I. BISHOP TINGLE.

Distribution of the Auxochromes in the Azo Compounds. H. KAUFFMANN AND W. Kugel. Ber., 44, 2386-9; cf. C. A., 1, 47.—Phenyldiazonium salts and resorcinol form p- and o-phenylazoresorcinols, the former being in larger quantity. Under benzoylresorcinol phenyl-p-azoresorcinyl similar conditions, gives benzoate. C18H18N2O2, only; yellow crystals from alc., m. 180°. Yield, good. Alkalies hydrolyze it to phenylazoresorcinol, m. 169°. Attempts to combine benzoylresorcinol, or resorcinyl methyl ether with diazotized "aminoresorcinyl dimethyl ether" were unsuccessful. 2,4-Dimethoxynitrobenzene is reduced to 2,4-dimethoxyaminobenzene hydrochloride by Sn and conc. HCl; long, pale blue needles from H<sub>2</sub>O. Yield, about 80%. When diazotized and treated with resorcinol it forms 2,4-dimethoxy-2',4'-dihydroxyazobenzene, (MeO)<sub>2</sub>C<sub>2</sub>H<sub>2</sub>N: NC<sub>2</sub>H<sub>2</sub>(OH)<sub>2</sub>; almost black crystals from toluene, m. 186°. It is brown if finely divided; in alkali the color is orange-yellow; in conc. HCl, dark red; in conc. H<sub>2</sub>SO<sub>4</sub>, dark blue. With Me<sub>2</sub>SO<sub>4</sub> and aq. NaOH (10%) it gives 2,4,2',4'-tetramethoxyazobenzene (MeO), C,HaN: NC,H3(OMe), red crystals from alc., m. 181°. 3,4,3',4'-Tetramethoxyazobenzene is prepared from 3,4-dimethoxynitrobenzene, Zn dust and alc. KOH; yellow crystals from alc., m. 163°. In HCl the color is carmosin-red. 2,5,2',5'-Tetramethoxyazobenzene was obtained in a similar manner

to the preceding compd.; m. 142°, not 140°. In conc. HCl, the color is blue; in conc. H<sub>2</sub>SO<sub>4</sub>, violet-red.

J. BISHOP TINGLE.

Oxidation of Allocinnamylideneacetic Acid. C. N. RIBER. Ber., 44, 2389-91.—At o°, in  $H_2O$ , KMnO<sub>4</sub> oxidizes K allocinnamylideneacetate, PhCH: CHCH: CHCO<sub>2</sub>K, to BzH and mesotartaric acid, which was isolated as the Ca salt. It is known that, under similar conditions, cinnamylideneacetic acid gives dl-tartaric acid, consequently the isomerism of cinnamylideneacetic and allocinnamylideneacetic acids must be due essentially to the nature of the  $\alpha,\beta$ -double linkage. The relationship of the parent acids must be the same as that of fumaric and maleic acids, because these are also oxidized to dl-tartaric and mesotartaric acids, resp. The suggestion which has been made so frequently, that maleic acid has the structure of a dihydroxylactone, is disproved by the above result, because the structure of maleic and allocinnamylideneacetic acids must be identical in this respect and the latter acid has only 1 acidic group.

I. BISHOP TINGLE.

 $\Delta^a$ -Butenylbenzene. C. N. RIBER. Ber., 44, 2391-3.— $\Delta^a$ -Butenylbenzene, PhCH<sub>2</sub>CH<sub>2</sub>CH: CH<sub>2</sub>, is prepared from Na wire (60 g.), abs. Et<sub>2</sub>O (300 cc.), allyl bromide (60 g.) and benzyl chloride (60 g.), at the ordinary temp., during a week; the Et<sub>2</sub>O soln. is removed and treated, under the same conditions, with fresh Na wire (40 g.) and at the end of a week this procedure is repeated. The Et<sub>2</sub>O soln. is washed with dil. HCl, then with H<sub>2</sub>O, dried with Na<sub>2</sub>SO<sub>4</sub> and distilled up to 120°, then under 10 mm. The portion b<sub>10</sub> 60-70° is further fractionated. Mobil, highly refractive liquid with a sharp odor, unlike that of its isomers, b<sub>10</sub> 64°, d<sub>4</sub><sup>20</sup> 0.8831,  $n_{20}^{20}$  1.5059, mol. ref. 44.44. In acetone, powdered KMnO<sub>4</sub> oxidizes the hydrocarbon to hydrocinnamic acid. The compds. hitherto described as  $\Delta^a$ -butenylbenzene are either impure, or are the  $\Delta^a$ - or  $\Delta^a$ -isomers.

Barium Oxide as a Reducing Agent: Reduction of Nitrobenzene to Nitrosobenzene. Azobenzene, Aniline, Phenazine and Ammonia. Th. Zerevitinov and I. Ostrom-ISSLENSKII. Chem. Lab., Imp. Techn. Hochsch., Moscow. Ber., 44, 2402-9.-At 208°, during 1.5-2 hrs., BaO (80 g.) and PhNO, (200 g.) form PhNO. When the PhNO<sub>2</sub> is dropped into a tube containing BaO, at 225-30°, the products consist of azobenzene, PhNH<sub>2</sub>, NH<sub>2</sub> and phenazine, which were fully identified. Yield, 28.4, 3.4, very little and 2.7% of the PhNO2, resp. At about 250°, Ba(OH)2 reduces PhNO2 to PhNH, and phenazine. At 265-70°, BaO and o-nitrotoluene give o-toluidine only; m-nitrotoluene is not changed under similar conditions, but if the temp. be raised it chars. p-Nitrotoluene and BaO, at about 270-80°, is reduced to p-toluidine and a little azo-p-toluene. The use of charcoal, purnice, CaO, or SrO, in place of BaO, causes no change in the PhNO, or in the nitrotoluenes, even at 255° and with the time of contact increased. Nitromethane, at 250-65°, is reduced by BaO, to NH<sub>a</sub>, MeNH<sub>a</sub> hydrocarbons of the CH<sub>4</sub> series and other substances. Phenazine and PhNH<sub>2</sub> are the only products of the interaction of PhNO, and barytes, under the conditions described above. The reducing action of BaO is ascribed to the formation of BaO, which is immediately decomp. into BaO + O. The O is not evolved, even in traces, it must be consumed in oxidizing a portion of the PhNO<sub>2</sub>. Phenazine hydroquinol, orangeyellow needles, chars slowly, m. 232° (decomp.). Phenazine resorcinol, light yellow needles, m. 213.5°. Phenazine pyrocatechol, stellate aggregates of pale yellow needles, m. 184°. The preceding 3 compds. have the formula, (HO)<sub>2</sub>C<sub>2</sub>H<sub>4·2</sub>C<sub>12</sub>H<sub>2</sub>N<sub>2</sub>; they are prepared by mixing alc. solns. of the components, at the ordinary temp. J. B. T.

Homology of Anthranil and Methylanthranil. JOHANNES SCHEIBER. E. Beckmann's Lab. Appl. Chem., Univ. Leipzig. Ber., 44, 2409–18.—Measurements have been made of the ultraviolet absorption spectrum of anthranil and of methylanthranil, the results being expressed in curves, constructed by plotting the thickness of the soln.

against the oscillation frequency, in the usual manner. The spectra are practically identical and are not affected by the solvent, alc.,  $Et_2O$ , hexane, alc. HCl (up to 100 equiv. of HCl), or aq. HCl (up to 4.9% of HCl). There is a difference at higher concs. (24.6% of HCl) which disappears in acid of 39% conc. The acid solns. undergo a change with time. Anthroxanic acid (anthranilcarboxylic acid) has practically the same absorption in alc. as in  $Et_2O$  and the curves are similar to those of anthranil and methylanthranil. These results indicate that the 3 compds. in question contain the same complex, (I) (R = H, Me or CO<sub>2</sub>H), that anthranil and methylanthranil are

$$C_{\bullet}H \stackrel{CR}{\swarrow_{N}} O \qquad C_{\bullet}H \stackrel{CO}{\swarrow_{NH}}$$

$$(II) \qquad (III)$$

homologs and that anthranil does not have the formula (II).

J. B. T.

o-Nitrophenylglyoxylic Acid. Gustav Heller. E. Beckmann's Lab. Appl. Chem., Leipzig. Ber., 44, 2418-21.—o-Nitrophenylglyoxylic acid is best prepared by dissolving o-nitromandelic acid (50 g.) in H<sub>2</sub>O (1 l.), and alkali, cooling the soln. and adding KMnO<sub>4</sub> (27 g.), in H<sub>2</sub>O (20 pts.). The soln. is allowed to remain 24 hrs. before filtering; the purified acid m. 156-7°, not 123°. It is best reduced to anthroxanic acid by dissolving in highly dil., aq. NH<sub>2</sub> and adding Zn dust and a little NH<sub>4</sub>Cl. Ethyl o-nitroglyoxylate, from the acid and b., alc. HCl; crystals from ligroin, or from C<sub>0</sub>H<sub>4</sub> + ligroin, m. 43-4.5°. Yield, 68% of the acid. With SnCl<sub>2</sub>, in conc. HCl, it gives ethyl anthroxanate; bundles of long needles from ligroin, m. 64-5°. Aq. NaOH hydrolyzes it to anthroxanic acid. Methyl anthroxanate, from the Ag salt and MeI, at the ordinary temp.; bundles of colorless crystals from ligroin, m. 70°. J. B. T.

Action of the Chlorides of Dibasic Aliphatic Acids on Sodioacetoacetic Ester. JOHANNES SCHEIBER. E. Beckmann's Inst. Appl. Chem., Univ. Leipzig. Ber., 44, 2422-9; cf. C. A., 3, 2165.—Succinyl chloride and Et sodioacetoacetate, m. Et.O. form ethyl β-carboxypropionylacetoacetate, HO2CCH2CH2COCHACCO2Et; prisms from Et.O. m. 82°. It gives a red color with FeCl, dissolves without decomp. in aq. NaOH and gradually decomp. spontaneously, at the ordinary temp., into succinic acid. Et acetoacetate and AcOH. With PhN, H, it gives the following 4 compds.: (1) Phenylhydrazine salt of ethyl β-carboxypropionylacetoacetatebisphenylhydrazone, C28H24O4N6; white, unstable crystals, m. 138°. It is formed in Et<sub>2</sub>O and it eliminates PhN<sub>2</sub>H<sub>2</sub> when treated with aq. NaOH, at the ordinary temp. (2) Pyrazole derivative, C18H18O4N2, is prepared in glacial AcOH, at the ordinary temp.; white crystals, m. 143°. It gives the pyrazoline reaction and is stable towards Fehling's soln. (3) A base, C14H14O2N2, or C22H22O4N4, is prepared like (2); white crystals from glacial AcOH, m. 157°. It resembles (2) in its reactions; mol. wt., in C<sub>n</sub>H<sub>a</sub>, 468; in glacial AcOH, 538. Chloroplatinate, (C14H14O2N2).H2PtCl6, yellow. (4) A compound, m. 214-5°. It is formed together with (3), is separated by Et<sub>2</sub>O and is sol. in alkali. With H<sub>2</sub>NOH, Et β-carboxypropionylacetoacetate forms an isoxazole, C<sub>10</sub>H<sub>12</sub>O<sub>2</sub>N, m. 81°. With N<sub>2</sub>H<sub>4</sub> the ester forms a hydrazone, C<sub>10</sub>H<sub>16</sub>O<sub>5</sub>N<sub>2</sub>; white crystals, m. 188°. It reduces Fehling's soln. A second compound, C10H14O4N2.2H2O is also formed from N<sub>2</sub>H<sub>4</sub> and is likewise produced from semicarbazine; m. 118°. It has acidic properties, has no enolic group and does not reduce Fehling's soln. A compound, possibly diethyl succinyldiacetoacetate, is formed together with the above propionyl ester, its quantity was small and it could not be purified. Diethyl glutaryldiacetoacetate, CH<sub>2</sub>(CH<sub>2</sub>COCHAcCO<sub>2</sub>Et)<sub>2</sub>, is prepared in a similar manner to the succinyl compd.; viscid oil; mol. wt., in C<sub>2</sub>H<sub>4</sub>, 309-38. The ester has acidic properties, it dissolves in Na<sub>2</sub>CO<sub>3</sub>, gives an intense reaction with FeCl<sub>3</sub> (? color) and is quickly decomp. by H<sub>2</sub>O, but is stable when dry. The chief product of its interaction with PhN.H. is glutaric-

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bisphenylhydraside, CH<sub>2</sub>(CH<sub>2</sub>CONHNHPh)<sub>2</sub>, which was also prepared from glutaric acid; white, lustrous plates from alc., or glacial AcOH, m. 217-8°. A compound is formed in very small quantity, together with the preceding phenylhydrazide, from the ester, m. 113-5°. It gives Knorr's reaction. Diethyl adipyldiacetoacetale, EtO<sub>2</sub>CCHAcCOCH<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>COCHAcCO<sub>2</sub>Et, is prepared like the glutaryl compd. which it resembles closely; mol. wt., in C<sub>2</sub>H<sub>4</sub>, 346-53; in glacial AcOH, after 16 hrs., 217. With PhN<sub>2</sub>H<sub>4</sub> it forms adipiciphenylhydraside, PhNHNHCOCH<sub>4</sub>(CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>-CONHNHPh, which was also prepared from adipic acid; pearly lustrous plates from alc., or glacial AcOH, m. 206-7°. A compound is formed in small quantity, together with the preceding hydrazide, from the ester; m. 250°. It gives a dull green color with alc. and FeCl<sub>4</sub>.

J. BISHOP TINGLE.

Thermochemical Investigations. IV. Diazo and Azo Compounds. V. SVYEN-TOSLAVSKII. Luginin's Thermal Lab., Moscow. Ber., 44, 2429-37; cf. C. A., 4, 2801.—The following results were obtained by the use of solid diazonium hydroxides, prepared by different methods. The reactions which have been studied are best expressed by equations; s = soln.; l = liquid. N: NPhOH(s) + HCl(s)  $\longrightarrow$  H<sub>2</sub>O(l) + N: NPhCl(s) + 11.68K. The value found previously was 13.70K; the present determinations are the more accurate.  $o\text{-MeC}_sH_sN(:N)OH(s) + HCl(s) \longrightarrow$  $H_*O(l) + o$ -MeC<sub>2</sub> $H_*N(: N)Cl(s) + 12.06K.$  p-MeC<sub>2</sub> $H_*N(: N)OH(s) + HCl(s) \longrightarrow$  $H_2O(l) + p-MeC_0H_4N(:N)Cl(s) + 13.00K.$  PhN(:N)Cl(s) + NaOH(s)  $\longrightarrow$ NaCl(s) + PhN(: N)OH(s) + 3.21K.  $p-MeC_2H_2N(: N)Cl(s) + NaOH(s) \longrightarrow$  $NaCl(s) + p-MeC_sH_sN(: N)OH(s) + 2.23K$ . The calculated value for o-MeC\_sH\_sN-(: N)Cl is 1.64K.  $(N : NPh)_sSO_4(s) + 2NaOH(s) \longrightarrow Na_sSO_4(s) + 2PhN(: N)$ OH(s) + 19.10K.  $p-[MeC_uH_aN(:N)]_sO_a(s) + 2NaOH(s) \longrightarrow Na_sO_a(s) +$  $2 p-MeC_2H_2N(: N)OH(s) + 18.29K.$   $0-[MeC_2H_2N(: N)]_SO_4(s) + 2 NaOH(s) Na_sSO_s(s) + 2$  o-MeC,  $H_sN(:N)OH + 20.28K$ . PhN(:N)OH(s) + HCl(s)  $\longrightarrow$ PhN(: N)Cl(s) +  $H_2O(l)$  + 10.08K. It follows that this last reaction does not consist simply in the neutralization of the base, but that some of the hydroxide must have change.  $PhN(: N)Cl(s) + \infty NaOH(s) \longrightarrow NaCl(s) + PhN:$ a  $NONa(s) + H_2O(l) + \infty NaOH(s) + 6.85K$ ; consequently N : NPhOH(s) +  $\infty$  $NaOH(s) \longrightarrow H_2O(l) + PhN : NONa(s) + _{\infty} NaOH(s) + 4.83K (= 6.85-2.02).$  $\rho$ -MeC<sub>2</sub>H<sub>2</sub>NCl : N(s) + 2 NaOH(s)  $\longrightarrow$  NaCl(s) + H<sub>2</sub>O(l) +  $\rho$ -MeC<sub>2</sub>H<sub>2</sub>N : NONa(s) + 3.96K. p-MeC<sub>2</sub>H<sub>4</sub>NCl : N(s) + 3 NaOH(s)  $\longrightarrow$  NaCl(s) + H<sub>2</sub>O(l) + NaOH(s) +  $p-MeC_aH_aN: NONa(s) + 4.05K.$   $p-MeC_aH_aNC1: N(s) + \infty NaOH(s) \longrightarrow NaCl(s)$ +  $H_4O(l)$  +  $\infty$  NaOH(s) + p-MeC<sub>2</sub> $H_4N$ : NONa(s) + 5.34K. PhNC1 : N(s) +  $\beta$ -C<sub>10</sub>H<sub>2</sub>ONa(s)  $\longrightarrow$  NaCl(s) + PhN : NC<sub>10</sub>H<sub>2</sub>OH (solid) + 30.77K. Cf. following J. BISHOP TINGLE. abstr.

Thermochemical Investigations. V. Diazo and Azo Compounds Monoamines. V. SVYENTOSLAVSKII. Polytechnic. Kiev. Ber., 44, 2437-45; cf. preceding abstr.— A continuation of (C. A., 4, 2488); the letters in the equations have the same significance as above.  $p\text{-MeC}_0H_4NH_2.HCl(s) + HNO_2(s) \longrightarrow 2 H_2O(l) + p\text{-MeC}_0H_4NCl \longrightarrow NCl: N(s) + 23.72K. <math>p\text{-MeC}_0H_4NCl: N(s) + 4 NaOH(s) \longrightarrow p\text{-MeC}_0H_4NCl: N(s) + 6\text{-}C_10H_2ONa(s) \longrightarrow NaCl(s) + p\text{-MeC}_0H_4N: NC_10H_0OH (solid) + 29.12K. <math>p\text{-MeC}_0H_4NCl: N(s) + 20.12K. p\text{-MeC}_0H_4NCl: N(s) + 24.55K. p\text{-MeC}_0H_4NCl: N(s) + 24.55K. p\text{-MeC}_0H_4NCl: N(s) + 24.55K. p\text{-MeC}_0H_4NCl: N(s) + 24.55K. p\text{-MeC}_0H_4NCl: N(s) + 3.87K. p\text{-MeC}_0H_4NCl: N(s) + 6\text{-}C_10H_2ONa(s) \longrightarrow NaCl(s) + p\text{-MeC}_0H_4NCl: N(s) + 3.87K. p\text{-MeC}_0H_4NCl: N(s) + 4.47K. p\text{-MeC}_0H_4NCl: N(s) + 4.47K. p\text{-MeC}_0H_4NCl: N(s) + 4.47K. p\text{-MeC}_0H_4NCl: N(s) + 24.55K. p\text{-MeC}_0H_4NCl: N(s) + 4.47K. p\text{-MeC}_0H_4NCl: N(s) + 24.55K. p\text{-MeC}_0H_4NCl: N(s) + 4.47K. p\text{-MeC}_0H_4NCl: N(s) + 4$ 

NONa(s) + 12.66K. N : NCIC<sub>2</sub>H<sub>4</sub>C<sub>3</sub>H<sub>4</sub>NCl : N(s) + 2  $\beta$ -C<sub>10</sub>H<sub>7</sub>ONa(s)  $\longrightarrow$  2 NaCl(s?) + HOC<sub>10</sub>H<sub>4</sub>N : NC<sub>2</sub>H<sub>4</sub>C<sub>3</sub>H<sub>4</sub>N : NC<sub>10</sub>H<sub>6</sub>OH (solid) + 59.83K. HCl.NH<sub>2</sub>-C<sub>3</sub>H<sub>4</sub>C<sub>4</sub>H<sub>4</sub>NH<sub>2</sub>.HCl(s) + 2 NaOH(s)  $\longrightarrow$  2 NaCl(s) + 2 H<sub>2</sub>O(l) + H<sub>2</sub>NC<sub>2</sub>H<sub>4</sub>C<sub>3</sub>H<sub>4</sub>NH<sub>2</sub> (solid) + 21.1qK.  $\rho, \rho'$ -HCl.NH<sub>2</sub>C<sub>3</sub>H<sub>4</sub>(OMe)C<sub>3</sub>H<sub>4</sub>(OMe)NH<sub>2</sub>.HCl(s) + 2 HNO<sub>2</sub>(s)  $\longrightarrow$  4 H<sub>2</sub>O(l) + N : NClC<sub>4</sub>H<sub>4</sub>(OMe)C<sub>4</sub>H<sub>6</sub>(OMe)NCl : N(s) + 46.35K.  $\rho, \rho'$ -N : NCl-C<sub>4</sub>H<sub>6</sub>(OMe)C<sub>4</sub>H<sub>6</sub>(OMe)NCl : N(s) + 2 H<sub>2</sub>O(l) + 4 NaOH(s) + NaON : NC<sub>4</sub>H<sub>6</sub>(OMe)C<sub>4</sub>H<sub>6</sub>(OMe)N : NONa(s) + 14.07K.  $\rho, \rho'$ -N : NCl-C<sub>4</sub>H<sub>6</sub>(OMe)C<sub>4</sub>H<sub>5</sub>(OMe)NCl : N(s) + 2  $\beta$ -C<sub>10</sub>H<sub>7</sub>ONa(s)  $\longrightarrow$  2 NaCl(s) + HOC<sub>10</sub>H<sub>4</sub>N: NC<sub>4</sub>H<sub>6</sub>(OMe)C<sub>4</sub>H<sub>5</sub>(OMe)N : NC<sub>10</sub>H<sub>6</sub>OH (solid) + 60.56. These diazotizations were carried out in acidified, aq. soln. The results given above and also the values obtained previously for similar reactions are tabulated and discussed. J. B. Tingle.

New Reactions of Dicyanogen and of the Acyl Cyanides. D. VORLÄNDER. Chem. Inst., Univ. Halle a/S. Ber., 44, 2455-76.—Contrary to the statement of Desgrez (Bull. [3] 13, 735) benzoyl cyanide, BzCN, is produced by the interaction of CaHa, AlCl, (equal parts) and C,N, the liquid is cooled with H,O. Yield, 4% of the C,H,. When the C<sub>2</sub>H<sub>2</sub> is b. the product consists of benzonitrile and benzoyl cyanide. Yields, 9 and 18% of the C.H. resp. At 40°, during 4 hrs., the yields of the cyanide and nitrile are 24 and 6% of the C.H., resp. The first product of the reaction is supposed to be the hypothetical imine, HN: CPhCN, a part of this is hydrolyzed to BzCN by the dil. acid employed in the separation of the products of the reaction, a portion decomp. spontaneously into HCN + PhCN and a portion combines with C.H. to form the hypothetical diimine, HN: CPhCPh: NH, which is hydrolyzed to benzil. The other products of the reaction, which have been isolated, are as follows: BzOH; a blue compound, which gives a colloidal soln. in H<sub>2</sub>O and is quickly decomp. by HCl, or by b. alkali hydroxides; a compound, colorless needles from acetone, becomes green 150°, gradually becomes blue, m. 188-90°. p-Methylbenzoyl cyanide (7.2), p-methylbenzonitrile (8), p,p'-dimethylbenzil (10) and p-toluylic acid (3.4) were obtained from toluene, at 40°, during 6 hrs., under the conditions described above. The numbers in parentheses refer to the resp. yields in % of the toluene. The chief product of the interaction of ethylbenzene (30 g.), AlCl<sub>2</sub> (20 g.) and C<sub>2</sub>N<sub>2</sub> consisted of an oil, which could not be distilled; in addition there are formed ethylbenzoyl cyanide, isolated as the anilide (1 g.), p-ethylbenzonitrile (5.5 g.), p-ethylbenzoic acid (1.7 g.) and a compound (3 g.); pale yellow needles from acetone + H<sub>2</sub>O, m. about 185°, previously softening. Biphenyl, in CS<sub>2</sub>, reacts only to a very limited extent with AlCl<sub>2</sub> and C<sub>2</sub>N<sub>2</sub>, the product was p-phenylbenzoyl cyanide (isolated as the anilide, after hydrolysis). p-Ethoxybenzoyl cyanide, is prepared by distilling p-ethoxybenzoyl chloride with Hg(CN)<sub>2</sub> in excess; prismatic plates from petroleum ether, m. 43°. Yield, 75%. It is also formed from ethoxybenzene, (20 g.) CS<sub>2</sub>, (40-50 g.) AlCl<sub>2</sub> (50 g.), C<sub>2</sub>N<sub>2</sub> and dry HCl. Yield, 22.5% of the ethoxybenzene. In conc. H<sub>2</sub>SO<sub>4</sub> the color is green, changing to blue. B., dil. HCl hydrolyzes it to p-ethoxybenzoic acid. When fused with PhNH, the cyanide gives p-ethoxybenzanilide. p-Ethoxyphenylglyoxylic acid is prepared from the cyanide and cone. HCl, at the ordinary temp., during 3 days; colorless, hydrated prisms from H<sub>2</sub>O, m. 52°, anhydrous crystals from C<sub>2</sub>H<sub>4</sub>, m. 125° (gas evolution). It reduces Ag-NH<sub>2</sub> soln. and gives the following derivatives: phenylhydrazone, long, yellow needles from dil. alc., m. 153° (decomp.). Azinecarboxylic acid, from N<sub>2</sub>H<sub>4</sub>; pale yellow needles from alc., m. 173-6° (CO<sub>2</sub> evolution). An acid, from HONH<sub>2</sub>; m. 152-4° (decomp.). p,p'-Diethoxybensil, is prepared in the manner described above for p-ethoxybenzoyl cyanide, but at the end of 4 hrs. fresh AlCla (30 g.) is added and the treatment with C<sub>2</sub>N<sub>2</sub> + dry HCl is continued during 4 hrs. the product being then allowed to remain in a closed vessel during 3-4 days; long, allvery white, microscopic prisms from Et<sub>2</sub>O + ale., m. 149°. Yield, 5% of the ethoxy-

benzene. In conc. H.SO, the color is red. Osazone, from PhN.H., in giscial AcOH; small, yellow plates, m. 171°. On exposure to light and air it becomes yellowish red, but regains its light yellow color when heated. p-Methoxybenzoyl cyanide is obtained by the methods described for the ethoxy compd. 9-Cyanofluogene and a little benzil are formed from benzoyl cyanide, C.H., AlCl, and dry HCl, at the ordinary temp., but unless the AlCl, be recently prepared the products are resins and oils. Benzoyl cyanide (1 mol.), C<sub>2</sub>H<sub>4</sub> (2 mols.), CS<sub>2</sub> and AlCl<sub>2</sub> when heated give triphenylacetonitrile. At oo, toluene, AlCl, and benzoyl cyanide form di-p-tolylphenylacetonitrile, it is also produced at 50-60°, but the proportions of resin and oils are larger; monoclinic plates from Et<sub>2</sub>O, or acetone + H<sub>2</sub>O, m. 132-3°; mol. wt., in C<sub>2</sub>H<sub>4</sub>, 281-92. Yield, 123% of the cyanide. The compd. is also obtained by heating di-p-tolylphenylmethyl chloride and Hg(CN). In a similar manner, di-p,p'-ethyltriphenylacetonitrile is obtained from ethylbenzene, CS<sub>2</sub>, AlCl<sub>2</sub> and benzoyl cyanide; colorless plates from acetone + H<sub>2</sub>O, m. 111-2°. Di-p,p'-methoxytriphenylacetonitrile, from methoxybenzene and benzoyl cyanide; short rods from MeOH, m. 98°; mol. wt., in C<sub>2</sub>H<sub>2</sub>, 340°. Yield, about 214% of the cyanide. In conc. H.SO, the color is orange-red. The compd. is not changed by alc. KOH at 150°, but is decomp. by fused KOH, or by fuming HCl at 200°. Alc. and Na reduce it to di-p,p'-methoxytriphenylmethane. The cyanide is also formed in small quantity from di-p,p'-methoxytriphenylchloromethane and Hg(CN)<sub>2</sub>. Di-p,p'-hydroxytriphenylacetonitrile is prepared by b. the above dimethoxy compd. with aq. HI (d. 1.7); hexagonal plates from Et<sub>2</sub>O + ligroin, m. 202°. In conc. H,SO, the color is orange-red; with alc. + FeCl, pale brownish red. It regenerates the dimethoxy compd. by treatment with aq. alkali hydroxide and MerSO4. Diacetyl derivative, from Ac O; colorless, triclinic plates from MeOH + H.O, m. 130°; mol. wt., in C<sub>2</sub>H<sub>a</sub>, 378. Prolonged b. of di-p,p'-methoxytriphenylacetonitrile with aq. HI (d. 2.0) and red P converts it into p-hydroxydiphenylacetic acid. Methyl ester, oily. Ethyl ester, from abs. alc. and conc. H<sub>2</sub>SO<sub>4</sub>; stellate prisms from CS<sub>2</sub>, m. 92°; mol. wt., in CaHa, 268. p-Methoxytriphenylacetonitrile is formed from p-methoxybenzoyl cyanide and C.H., white needles from MeOH, m. 137°; mol. wt., in C.H., 285. Yield, 54% of the cyanide. In conc. H<sub>2</sub>SO<sub>4</sub> the color is violet. Di-p,p'-ethoxytriphenylacetonitrile, from benzoyl cyanide and ethoxybenzene; short, pointed prisms from alc., m. 120°. In conc. H2SO4 the color is orange-red. With HI it gives the same compds. as does the above methoxy nitrile. In absence of air, C<sub>2</sub>N<sub>2</sub>, SO<sub>2</sub> and H<sub>2</sub>O combine, at the ordinary temp., during 2-3 hrs., forming chiefly HCN and H2SO4. together with NH, salts. I. BISHOP TINGLE.

Fermentation of Pyruvic and Oxalacetic Acids as a Lecture Experiment. C. Neuberg and L. Karczag. Chem. Dept., Vet. Inst. Roy. Agric. Hochsch., Berlin. Ber., 44, 2477-9.—Aq. solns. (1%) of pyruvic and of oxalacetic acids are readily fermented by yeast (2 g.). The reactions are carried out in tubes, immersed in H<sub>2</sub>O, at 38-40%. Evolution of CO<sub>2</sub> is easily observed after 5 mins. The other product consists of AcH, which may be detected in the usual manner. The alkali salts of the acids react like the free acids; the progress of the change, in this case, can be shown by the liquid, which was originally neutral, becoming strongly alkaline owing to the production of alkali carbonate.

J. BISHOP TINGLE.

Constituents of Coal. Ame Picter and Louis Ramseyer. Org. Lab., Univ. Geneva. Ber., 44, 2486-97.—A "fat" French gas coal, from Montrambert (Loire) when extracted with  $C_6H_6$  gave a hexahydrofluorene,  $C_{10}H_{10}$ , together with some polymers, chiefly  $(C_{13}H_{10})_2$ . They were separated by fractional distillation; oily liquid, b. 240-50°,  $b_{10}$  110-20°,  $d_{20}$  0.920, mol. wt., in freezing ethylene bromide, 172°. In  $C_6H_6$  it has a violet fluorescence. When b. the hydrocarbon polymerizes; at a red heat it is converted into H and fluorene. With Br the hydrocarbon gives bromo- and

dibromofluorene. Aq. KMnO<sub>4</sub> (1%) and NaOH oxidize the hydrocarbon to AcOH, adipic and oxalic acids, showing that it has formula (I) or (II) below. Conc. HNO<sub>4</sub>

converts the hydrocarbon into a mixture of amorphous oxidation and nitration products; with fuming HNO<sub>2</sub> (3 parts), conc. H<sub>2</sub>SO<sub>4</sub> (3 parts) and glacial AcOH (5 parts) the hydrocarbon (1 part) gives a dinitrotetrahydrofluorenone, C<sub>12</sub>H<sub>10</sub>O<sub>4</sub>N<sub>2</sub>; light yellow, amorphous powder from alc. + H<sub>2</sub>O, decomp. without m. 95-100°. Oxime, colorless, decomp. without m. The ketone is reduced by Sn + HCl to diaminotetrahydrofluorenol; colorless solid darkens, in air. It gives the reactions of a metadiamine. Dibensoyl derivative (III), by the Schötten-Baumann method; crystals from dil. AcOH,

or alc., m. 150°. Distillation of the above coal (10 mm.) up to a temp. not exceeding 450°, gives a mixture of alicyclic hydrocarbons including (I) or (II). This is stated to be the better method for its production.

J. BISHOP TINGLE

Derivatives of 1,2-Dimethylbenzene. II. EMIL DIEPOLDER. Munich. Ber., 44, 2498-503; cf. C. A., 3, 2941.—The yield of 1,2-dimethyl-3,4-benzoquinone-3-phenylhydrazone, from 4-hydroxy-1,2-dimethylbenzene and phenyldiazonium sulfațe, in pyridine, is 22%. A similar result was obtained by the use of more than 1 mol. of aq. NaOH, at about -2°. Under these conditions the formation of resin was especially small. N-Acetyl-5-amino-4-hydroxy-1,2-dimethylbenzene, C10H13O2N, is prepared by mixing the aminohydroxide with Ac<sub>2</sub>O, allowing the mixture to become warm, dissolving the product in dil., aq. NaOH and adding AcOH, or by acetylation in presence of pyridine; aggregates of thin plates from alc., or rectangular plates from dil. alc., softens 184°, m. 190.5-1°. With alc. and FeCl, its color is green. At about 220° it decomp. into H<sub>2</sub>O and ethyleneaminohydroxyxylene. O,N-Diacetyl-5-amino-4-hydroxy-1,2-dimethylbenzene, C12H14O2N, is prepared by dissolving the aminohydroxyxylene in a mixture of Ac<sub>2</sub>O (5 parts) and pyridine (5 parts); slender, white, silky lustrous, radial aggregates of needles, or thick, almost colorless, quadratic, or octagonal prisms, m. 156-7°, sublimes 121°. It gives no color with FeCl.; cold, dil., aq. NaOH hydrolyzes it to the above monoacetyl compd. Triacetyl-5-amino-4-hydroxy-1,2-dimethylbenzene, C14H17O4N, by b. the aminohydroxyxylene with Ac4O + AcONa, during 3 hrs.; colorless, hexagonal plates of the monoclinic system from alc., m. 100.5-1.5°. When b. with H<sub>2</sub>O it is hydrolyzed to the preceding compd.; cold, aq. NaOH converts it into the above mono-Ac deriv. Triacetyl-o-aminophenol, C12H12O4N, is prepared in a similar manner to the preceding compd., from o-aminophenol; long, thin, colorless needles from petroleum ether, m. 78-9°. Warm H<sub>2</sub>O hydrolyzes it to the di-Ac compd., more prolonged b. converts it into the N-mono-Ac deriv. Aq. SO, reduces 1,2-dimethyl-4,5-benzoquinone to 4,5-dihydroxy-1,2-dimethylbenzene. Treatment of it with Ag<sub>2</sub>O failed to give a colorless modification of 1,2-dimethyl-4,5-benzoquinone (peroxide). The red and yellow crystals described previously have been measured,

they are crystallographically identical. The quinone colors the skin brown, decomp. spontaneously after about 7 months, reacts violently with N<sub>2</sub>H<sub>4</sub>.H<sub>4</sub>O, liberates I and gives a blue color with guaiacol resin in soln.

J. BISHOP TINGLE.

Quantitative Investigations of the Sulfonation of Toluene. A. F. HOLLEMAN AND P. CALAND. Wyk aan Zee. Ber., 44, 2504-22,-o-Toluenesulfonic acid is best obtained pure by cryst. the Ba salt, which is more readily sol. in H<sub>2</sub>O than that of the p-acid. Under the microscope the o-salt appears as granules, the p-salt as long, slender needles. p-Toluenesulfonic acid is best prepared from p-toluenesulfonyl chloride and H<sub>2</sub>O. m-Toluenesulfonic acid is most readily obtained from m-thiocresol and aq. "permanganate." Tables are given showing the freezing points of various mixtures of the isomeric sulfonyl chlorides. The quant. results given below are based on these determinations. Studies have been made of the influence, on the sulfonation of toluene, of temp. and of the conc. and quantity of the acid employed. As the temp. increases the proportion of o-acid diminishes; at 75° the production of p-acid reaches a maximum (p-, 75%; m-, 19%; o-, 6%). The conc. of the acid (96-100% H<sub>2</sub>SO<sub>4</sub>) is without influence on the results. The sulfonation is incomplete by the use of 1 or 2 parts of H<sub>2</sub>SO<sub>4</sub>; with 6 parts of acid all the toluene reacts except at o°. The proportion of p-acid is increased about 10% by the use of smaller proportions (not exceeding 6 parts) H<sub>2</sub>SO<sub>4</sub> at 0°; at 35° this influence is not operative and at 75° or 100°, with 41.5 parts of H<sub>2</sub>SO<sub>4</sub>, the yield of p-acid is increased about 4%, the actual ratio, under these conditions, being 78.5, 6.2 and 15.3% at 75° and 81.7, 14.4 and 3.9% at 100° of \$-\$. m- and o-acids, resp. The depression of the freezing ps. of the mixtures of the oand p-sulfonyl chlorides, which were obtained in the course of the above expts., is ascribed to the presence of m-toluenesulfonyl chloride. Special efforts were made to show that the depression was not caused by the presence of P compds., of toluenedisulfonyl chlorides, or of those of phenyl-, or xylenesulfonic acids. At 35°, toluene and HSO<sub>2</sub>Cl gave 58.8, 3.8 and 37.5% of p-, m- and o- acids resp., the corresponding results, by the use of H<sub>2</sub>SO<sub>4</sub>, being 63.1, 6.3 and 30.6%, resp. The addition of Ag<sub>2</sub>SO<sub>4</sub>, K,SO, or Hg,SO, to the H,SO, has no special influence on the ratio of the isomeric sulfonic acids. This simply confirms Holdermann's results (Ber., 39, 1252 (1906)). At 100°, m-toluenesulfonic acid is not changed by the prolonged action of H.SO. At 75°, under similar conditions, there is a slight reciprocal conversion of the o- and p-acids, but all 3 acids are formed directly from toluene and H<sub>2</sub>SO<sub>4</sub> and any subsequent change of o- p- which may occur, takes place directly and does not have toluene as an intermediate product. J. BISHOP TINGLE.

Constitution of the Aliphatic Diazo Compounds and of Hydrazoic Acid. JOHANNES THIELE. Chem. Inst., Univ. Strassburg. Ber., 44, 2522-5.—Certain products formed by the interaction of N<sub>2</sub>H<sub>4</sub> and ketones should be formulated as hydrazones, H<sub>2</sub>NN: CMeCO<sub>2</sub>R, and not as hydrazo compds. formula (I). The substances in question are

$$\begin{array}{c|c} HN \\ | \\ HN \end{array} \text{CMeCO}_2R \qquad O: C \overbrace{\hspace{1cm}} C: N: N \\ \hline (II) \\ \hline (III) \\ \hline \end{array}$$

oxidized to diazo (diazonium) derivatives, N: N: CRR', which cannot contain a cycloid of 3 members. The aliphatic diazo compds. are formulated as >C: N: N and their primary HCl addition products as >CHNCl: N. Quinonediazides are regarded as being (II) and (III) and nitrous oxide and hydrozoic acid as O: N: N and HN: N: N, resp. Pentavalent N ats., which are not ionizable, are generally assumed to exist in the nitro compds. Many of the known reactions of the substances in question are in better accord with the new formulas than with those in current use. Further experimental evidence will be published later. J. Bishop Tingles.

Primary Dinitro, Nitronitrite and Dialdoxime Compounds of the Aliphatic Series. J. v. Braun and W. Sobecki. Chem. Inst., Univ. Breslau. Ber., 44, 2526-34. In Et<sub>2</sub>O, 1,4-diiodobutane and solid AgNO, give a mixt., which is sepd. by distillation (15 mm.). The portion b. 60-105° consists of butane dinitrite, ONOCH, (CH.), CH.ONO. Yield, small. The 2nd fraction (105-20°) consists of nitrobutane nitrite, O2NCH2-(CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>ONO (see below). Yield, 20-5%. The 3rd portion (160-87°) is 1,4-dinitrobutane, O<sub>2</sub>NCH<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>NO<sub>2</sub>; almost colorless, nearly odorless, stable liquid, b<sub>13</sub> 176-8°. Yield, 50-5%. Sodium salt, from EtONa; small crystals. In H<sub>2</sub>O it gives white ppts. with AgNO, and Pb salts; a green ppt. with Cu salts and a deep red color with FeIII salts. In alk, soln., with Br, the dinitro compd. gives dinitrotetrabromobutane, O2NCBr2CH2CH2CBr2NO2; lustrous crystals from HCO2H, m. 100°. Dinitrolic acid, oily. Bisazo compounds, red, crystallize with difficulty. Nitrobutane nsitrite (see above) is unstable, b, about 110°. It is reduced by SnCl, in conc. HCl, to δ-hydroxybutylamine, HOCH<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>. Dibenzoyl deriv. m. 75° not 58° (Henry). The compds. described below were prepared in a similar manner to the preceding substances. Dinitropentane, (O2NCH2CH2)2CH2, from 1,5-diiodopentane; almost odorless, slightly colored liquid, b<sub>18</sub> 194-6°. Yield, about 55%. Disodium salt, cryst., explodes slightly when heated. Tetrabromo derivative, (O<sub>2</sub>NCBr<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>, crystals from HCO<sub>2</sub>H, m. 39°. Bisazo benzene derivative, (PhN<sub>2</sub>CH(NO<sub>2</sub>)CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub> from phenyldiazonium sulfate; red crystals from alc., m. 169°. Bisnitrolic acid, oil. Nitropentane nitrite, O2NCH2(CH2)2CH2ONO, unstable liquid, b12 130-3°. It is reduced to e-hydroxyamylamine, HOCH<sub>1</sub>(CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>; colorless liquid, b<sub>16</sub> 122°. It has an odor of fish. Chloroaurate, yellow needles, darkens on exposure to light. Benzoyl derivative, oily. The constitution of the hydroxyamine its shown by its conversion into piperidine by b. with conc. aq. HI, followed by treatment with aq. alkali. 1,10-Dinitrodecane, C<sub>10</sub>H<sub>20</sub>(NO<sub>2</sub>)<sub>2</sub>, from 1,10-diiododecane; crystals from Et<sub>2</sub>O + alc., m. 49°. Yield, 65%. Disodium salt. Tetrabromide, oil. The above Na salts, when added, at oo, to SnCl, (3 parts), in conc. HCl, are reduced to dioximes. The acid liquid is made slightly alk. with Na, CO, and extracted quickly with Et,O. In this manner dinitrobutane gives succinic dialdoxime. Glutaraldehyde dioxime, (HON: CHCH<sub>2</sub>)<sub>2</sub>CH<sub>4</sub>, is obtained from dinitropentane; m. 178°, not 171° (Harries). Yield, 55-60%. Decane dioxime, HON: CHCH2(CH2)6CH2CH: NOH, from dinitrodecane; crystals, softens 137°, m. 141°. J. BISHOP TINGLE.

Triphenylmethyl Peroxide. Contribution to the Chemistry of Free Radicals. HEINRICH WIELAND. Chem. Lab., Acad. Sci., Munich. Ber., 44, 2550-6; cf. C. A., 5, 3286.—Tetraphenyl-sym.-diphenoxyethane, PhOCPh, CPh, OPh, is formed by b. triphenylmethyl peroxide with xylene, in a CO, atm. entirely free from O; it is deposited when abs. alc. is added to the xylene; colorless, rhombohedral plates from C.H. softens and becomes yellow 135°, m. about 196°; in CO<sub>2</sub>, becomes yellow 150°, m. 215°; mol. wt. in C<sub>6</sub>H<sub>4</sub>, 494. In conc. H<sub>2</sub>SO<sub>4</sub> it slowly becomes yellow. Yield, 75%. B. with glacial AcOH + Zn dust converts it into PhOH, benzhydryl acetate and, in small quantity, a compound, possibly, PhOCPh<sub>2</sub>C<sub>2</sub>H<sub>4</sub>CHPhOPh. At 230-40°, in an atm. of CO<sub>2</sub>, tetraphenyl-sym.-diphenoxyethane decomp. into tetraphenylethylene, diphenyldiphenoxymethane, Ph<sub>2</sub>C(OPh), benzophenone and PhOH. Diphenyldiphenyloxymethane is sepd. by its solubility in alc.; vitreous, radial prisms from alc., m. 132°; mol. wt., in C<sub>2</sub>H<sub>4</sub>, 329. In conc. H<sub>2</sub>SO<sub>4</sub> the color reaction is similar to that of benzophenone; after adding H<sub>2</sub>O the liquid contains PhOH and benzophenone. In addition to the tetraphenyl-sym.-diphenyoxyethane, triphenylmethyl peroxide, under the conditions described above, gives some triphenylcarbinol, benzophenone and PhOH. J. BISHOP TINGLE.

Absorption Spectrum of Triphenylmethyl and of the Triphenylcarbinal Salts. Kurt H. Meyer and Heinrich Wieland. Chem. Lab, Acad. Sci., Munich. Ber., 44, 2557-9.—The absorption spectra have been detd. of the following compds. in soln., but the solvent is not mentioned, except that for the 2 sulfates it was conc. H<sub>2</sub>SO<sub>4</sub>: triphenylmethyl, p-tritolylmethyl, p-trichloro- and p-triiodotriphenylmethyl, triphenylcarboxyl chloride, diphenylphenoxymethyl, biphenylenediphenylmethyl, triphenylcarbonium sulfate and p-triiodotriphenylcarbonium sulfate. Solns. of triphenylmethyl in C<sub>6</sub>H<sub>6</sub>, acetylene tertachloride, or PhNO<sub>2</sub> do not conduct an elec. current. The spectra of the triarylmethyls mentioned above are all very similar and consist of sharply defined bands, which are clearly visible even when the diln. is so great as to obliterate the continuous absorption. The spectra resemble that of NO<sub>2</sub>. The yellow, carbinol salt solns. exhibit general absorption only. In this manner the radical and the ior R<sub>2</sub>C can be distinguished easily.

J. BISHOP TINGLE.

Dihydroterpenylamine. George Francis Morrett. Chem. Inst., Univ. Kiel. Ber., 44, 2560-5.—Chlorotetrahydrocarvylamine, formula (I) below, is prepared from

dihydrocarvylamine (II) and dry HCl, in Et<sub>2</sub>O, at 0°; long, white needles, m. 205°. At 140-5°, during 4 hrs., pyridine converts it into dihydroterpenylamine (III); color-

less liquid with a slight, characteristic odor,  $b_{16}$  96-100°;  $d_{16}^{16.5}$  0.8909;  $n_D^{16-5}$  1.49284;  $n_\alpha$  1.48906;  $n_7$  1.50749; mol. ref. 49.83. The amine absorbs CO<sub>2</sub> from the air and is optically inactive; it consists of 2 isomers which could not be sepd. completely. With the exception of the Bz compd., the derivs. of the  $\beta$ -isomer are the less sol., but the  $\alpha$ -amine is formed in the larger quantity. Picrate ( $\alpha + \beta - ?$ ) formed in Et<sub>2</sub>O; m. about 176°.  $\alpha$ -Benzoyl derivative, prepared in pyridine soln.; long, white needles from alc., m. 214°.  $\beta$ -Salts. Hydrochloride, flat, quadratic plates from H<sub>2</sub>O, m. about 235° (decomp.). Nitrate, rectangular plates from H<sub>2</sub>O, m. 179° (decomp.). Picrate, prepared in abs. Et<sub>2</sub>O; long, golden yellow needles from H<sub>2</sub>O, softens about 192°, m. about 195°. Benzoyl derivative, broad plates from alc., m. 178-9°.  $\beta$ -Dihydroterpenylamine,  $b_{16}$  100-1°. With HCl it regenerates (I). In glacial AcOH, O<sub>3</sub> oxidizes benzoyl- $\alpha$ -dihydroterpenylamine to acetone and 2-benzoylamino-1-methyl-4-cyclohexanone (IV). Under similar conditions benzoyldihydrocarvylamine (V) is

oxidized to i-methyl-2-benzoylamino-4-acetylcyclohexane (VI); white crystals from  $H_sO$ , m. 218-9°. J. Bishop Tingle.

Aniline Black and its Intermediate Compounds. ARTHUR GEORGE GREEN AND SALOMON WOLFF. Univ. Leeds, England. Ber., 44, 2570-9.—Mainly a reply to Willstätter and Cramer (C. A., 5, 3432). PhN<sub>2</sub>H<sub>2</sub> evolves N at 100°; consequently the N obtained by heating quinones, quinoneimines, or compds. of the aniline black series with PhN<sub>2</sub>H<sub>3</sub> can only be used as a measure of the reduction if the temp. employed does not exceed 90°. Willstätter and Cramer have heated their mixtures

with PhN<sub>2</sub>H<sub>2</sub> up to 150° and they assume that, even up to 200°, the spontaneous decomp. of PhN,H, is negligibly small. "Bichromate black," prepared according to the method of Willstätter and Dorogi, but omitting the treatment with acid, is identical with Green and Woodhead's emeraldine. It is a diquinoid, as shown by the action of PhN<sub>2</sub>H<sub>2</sub>, at 80-90°. The only impurity which could be found in it was a very little Cr(OH)<sub>2</sub>. "Purification" with acid causes a portion of the emeraldine to polymerize and a portion to decomp.; consequently W. and D.'s substances were mixtures. W. and C.'s statement that the Ti reduction method does not give the leuco bases, but only monoquinoids, is incorrect. The compds. in question are almost colorless and do not become lighter if the reduction takes place at 150°. Emeraldine and nigraniline forms true solns., but the presence of salts, or acids, even in small quantity. causes the formation of colloidal ppts. W. and C.'s results in this connection show that their substances were impure and they are also in error in stating that nigraniline and pernigraniline are not changed by dil. acids. Contrary to their statements, the nigraniline salts are blue. The authors' views on the oxidation products of PhNH. are summarized. J. BISHOP TINGLE.

Condensation Products of Isatin with Oxythionaphthene, Indandione and Indandone. E. NOELTING AND A. HERZBAUM. Chem. Schl., Mülhausen i-E. Ber., 44, 2585-90.—Thioquindolinecarboxylic acid, formula (I) below, is prepared by heating a mix-

$$\begin{array}{c|c}
\hline
c: N \\
\hline
co. c: C(CO_2H)
\end{array}$$
(II)
(II)

ture of isatin and oxythionaphthene with aq. NaOH, the resulting Na salt being subsequently acidified with AcOH; small, yellow needles from PhNO<sub>2</sub>, m. about 335°. It dyes wool and silk yellow in an acid bath and produces the same shades on cotton which has been treated with various mordants. Above its m. p. it decomp. into CO<sub>2</sub> and thioquindoline, m. 172°, not 169°. Quinolylenephenyleneketonecarboxylic acid (II), is obtained from indandione and isatin, in a similar manner to (I); slender, silky lustrous, white needles from xylene, m. about 340°. In presence of acid it dyes silk, wool and mordanted cotton pale yellow. At 350° (II) decomp. into CO<sub>2</sub> and quinolylenephenylene ketone. Quinolylenephenylenemethanecarboxylic acid (III), from

$$C: N \longrightarrow C: N \longrightarrow C: C(CO_2H) \longrightarrow C: N \longrightarrow C: C(CO_2H) \longrightarrow C: N \longrightarrow$$

indanone and isatin; pale yellow crystals from anisole, m. above 330° (decomp.). Yield, 85%. It has no affinity for animal or vegetable fibers. When m. (III) is converted into quinolylenephenylenemethane, which is also formed from indanone, o-aminobenzaldehyde and dil. HCl. It has been shown previously that indoxyl and isatin condense, in presence of aq. NaOH, to a quindolinecarboxylic acid (IV). The following salts have been prepared in order to compare their colors. Salts of (IV): Aluminium, yellow; chromium, yellowish green; lead, yellowish orange; silver, brownish red; mercuric, reddish orange; cobalt, orange; nickel, yellowish green. Salts of (I): Sodium, white crystals; aluminium, yellow with a green tinge; chromium, violet soln.; lead, greenish light yellow; silver, yellow with a brown shade; mercuric, yellow; cobalt, red; nickel, greenish. Salts of (II): Sodium, white crystals; aluminium, lead and mercuric, white; chromium, violet soln.; silver, reddish brown; cobalt, no. ppt.; nickel, pale greenish yellow. Salts of (III): Sodium, white crystals; aluminium,

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lead, silver and mercuric, white; chromium, violet soln.; coball, pale red; nickel, greenish yellow.

J. BISHOP TINGLE.

Degradation of Nerol and its Constitution. A. Blumann and O. Zhitschel. Anton Deppe Sohne's Lab., Hamburg-Billwärder. Ber., 44, 2590-3.—It has been suggested previously (Ber., 39, 1780 (1906)) that nerol is a stereoisomer of geraniol. This conclusion is confirmed by the fact that, when oxidized by aq. KMnO $_{\phi}$ , at o°, geraniol and nerol yield the same relative quantities of the same products, vis., acetone and levulic acid, isolated as the p-bromophenylhydrazone and the phenylhydrazone, resp. The acid was also identified in the free state.

J. BISHOP TINGLE.

δ-Phenyl Valeryl Ketone and δ-Phenylvaleric Acid. W. BORSCHE. Inst. Gen. Chem., Iniv. Göttingen. Ber., 44, 2594-6.—Methyl δ-phenylbutyl ketone, PhCH<sub>2</sub>-CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>C, is obtained by the action of H and colloidal Pd on cinnamalacetone, in MeOH; water-clear, highly refractive oil with a characteristic sweet odor, b<sub>74</sub> 268-9°. Yield, almost quant. Oxime, colorless, rather viscid liquid, b<sub>12</sub> 179-80°. With alk. aq. NaOBr, at 0°, the ketone is decomp. into CHBr<sub>2</sub> and δ-phenylvaleric acid, PhCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>H; thick, water-clear, rhombic plates from ligroin, m. 61°. Yield, 35% of the ketone. Methyl ester, colorless oil, b<sub>22</sub> 173°.

J. BISHOP TINOLE.

Condensation of p- and o-Methoxymandelic Nitrile with Phenois and Phenolic Ethers. A. BISTRZYCKI, J. PAULUS AND R. PERRIN. I. Chem. Lab., Univ. Freiburg, Switzerland. Ber., 44, 2596-617; cf. C. A., 5, 3262 and Ber., 31, 2821 (1898).— Anisaldehydecyanohydrin is best prepared by mixing the cryst. NaHSO, deriv. with H<sub>2</sub>O and highly conc. aq. KCN, at o°; long, colorless prisms from Et<sub>2</sub>O + petroleum ether, m. 57-8°, not 66-7°. Yield, 61%. 4-Hydroxy-4'-methoxydiphenylacetonitrile, MeOC\_H\_CH(CN)C\_H\_OH, is obtained by warming the preceding cyanohydrin (1 mol.) with PhOH (2.33 mol.) and H<sub>2</sub>SO<sub>4</sub> (73%). It is identical with Stoermer's "o-hydroxyphenylanisylacetolactone" (loc. cit.). Yield, about 90%. 4-Acetoxy-4'methoxydiphenylacetonitrile is prepared, as are the Ac derivs. mentioned below, by b. the resp. parent compds. with Ac<sub>2</sub>O and anhydrous AcONa; lustrous plates from dil. alc., m. 85.5-6.5°. 4-Hydroxy-4'-methoxydiphenylacetic acid, MeOC<sub>2</sub>H<sub>4</sub>C(CO<sub>2</sub>H)-C<sub>8</sub>H<sub>4</sub>OH, is obtained by b. the nitrile with conc., alc. KOH, during 8 hrs.; microscopic needles from very dil. alc., m. 148.5-9.5°. Yield, 90%. In conc. H<sub>2</sub>SO<sub>4</sub> the color is orange, changing to brownish red when heated, evolves CO 60°, evolution ceases 170°, the total quantity being about 66.6% of 1 mol. Ammonium salt, lustrous, slender needles. 3-Methyl-4-hvdroxy-4'-methoxydiphenylacetonitrile, MeOC,H,CH-(CN)C<sub>6</sub>H<sub>2</sub>MeOH, from o-cresol and anisalcyanohydrin as above; long, flat, colorless prisms from 50% AcOH + HO, m. 142-3°, previously softening. Yield, 75%. Acetyl derivative, small prisms from alc. + H<sub>2</sub>O, m. 74.5-6°. 3-Methyl-4-hydroxy-4'methoxydiphenylacetic acid is formed by hydrolyzing the nitrile with alc. KOH; the other acids of this series are prepared in a similar manner, unless otherwise stated. Long, flat prisms from H<sub>2</sub>O, soften 127°, m. 128-9°. Yield, 85%. In conc. H<sub>2</sub>SO<sub>4</sub> the color is red, changing to reddish brown when heated, at 100-20° it evolves 66.6% of 1 mol. of CO. Silver salt, finely divided ppt. The product from anisalcyanohydrin and m-cresol was vitreous, but with p-cresol 2-hydroxy-5-methyl-4'-methoxydiphenylacetolactone formula (I), is produced. Aq. KOH (6%) hydrolyzes it to 2-hydroxy-5-

methyl-4'-methoxydiphenylacetic acid; bundles of colorless, microscopic prisms, m. 140° (decomp.). Yield, almost quant. In conc. H<sub>2</sub>SO<sub>4</sub> the color is violet, it darkens

when heated; evolves CO 125°, less than 44% of 1 mol. being formed up to 220°. Ammonium salt, bundles of microscopic needles. 2-Hydroxy-5-methyl-4'-methoxydiphenylacetamide, MeOC, H. CH (CONH,) C. H. MeOH, from the lactone and aq. NH,; bundles of slender needles from alc. + H<sub>2</sub>O, decomp. 137.5°. 2-Hydroxy-5-methyl-4'-methoxydiphenylacetohydrazide, MeOC, H, CH(CONHNH,)C, H, MeOH, from the lactone and N<sub>2</sub>H<sub>4</sub>. H<sub>2</sub>O, in dil. alc.; microscopic plates from alc. + H<sub>2</sub>O, decomp. 182.5°. It dissolves in cold, 1 N aq. KOH and in warm, aq. Na<sub>2</sub>CO<sub>2</sub> and therefore probably has an open chain formula. In conc. H.SO, the color is violet, it becomes deeper on adding a little K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>. Bensylidene derivative, MeOC<sub>2</sub>H<sub>4</sub>-CH(CONHN: CHPh)C.H.MeOH, from the preceding compd. and BzH, in alc., almost colorless, small, rectangualr plates, m. 184°. 3,4-Dihydroxy-4'-methoxydiphenylacetonitrile, MeOC<sub>2</sub>H<sub>4</sub>CH(CN)C<sub>4</sub>H<sub>4</sub>(OH)<sub>2</sub>, is prepared in a similar manner to the PhOH deriv., from pyrocatechol. The analogous compds. mentioned below are obtained in the same way, from the resp. phenols mentioned. Bundles of colorless needles from toluene, darkens 130°, m. 153.5-4.5°. Yield, of pure compd., 25%. In conc. H.SO, the color is blue, changing to bluish violet when warmed. Diacetyl derivative, plates from alc. + AcOH (50%), m. 77-8°. [2-Hydroxy-1-(?)naphthyl]-4'-methoxyphenylacetolactone (II), from anisalcyanohydrin and  $\beta$ -naphthol; long, colorless, prismatic needles from AcOH (50%), m. 145-6° (decomp.). Yield, of pure compd., 25%. In conc. H,SO, the color is green, changing to greenish blue when heated. No definit compd. could be obtained from  $\alpha$ -naphthol and anisalcyanohydrin, but with anisole the product consists of di-p,p'-anisylacetonitrile, (MeOC,H<sub>4</sub>),CHCN; long, lustrous prisms from alc., m. 154.5°. previously softening. Yield, 75%. In conc. H<sub>2</sub>SO<sub>4</sub> the color is faintly violet, it becomes more intense when warmed. The nitrile is hydrolyzed to di-p,p'-anisylacetic acid. Yield, 75%. Its color in conc. H<sub>2</sub>SO<sub>4</sub> is reddish violet, changing to brownish red when heated, CO is evolved about 60° and the evolution is most energetic at 90-105°. Ammonium sall, microscopic needles from aq. NH<sub>a</sub>Cl. It is unstable. Di-p,p'-anisylacetyl chloride, (MeOC<sub>a</sub>H<sub>a</sub>)<sub>a</sub>CHCOCl, from the acid, PCla and POCla; colorless prisms from ligroin, m. 58-61°. In a current of CO. it evolves CO + HCl at 80-235°, the quantities being 50 and 66.6% of 1 mol., resp. The residual solid could not be cryst. 4-Ethoxy-4'-methoxydiphenylacetonitrile, MeO-C.H.CH(CN)C.H.OEt, from ethoxybenzene; small, lustrous needles from alc., m. 87-8°. Yield, 44%. In conc. H<sub>2</sub>SO<sub>4</sub> the color is blue, changing when heated to violet, then to brown. 3,4,4'-Trimethoxydiphenylacetonitrile, MeOC<sub>2</sub>H<sub>4</sub>CW(CN)C<sub>2</sub>H<sub>2</sub>(OMe)<sub>2</sub>, veratrole; colorless, hexagonal prisms from MeOH, m. 96°, previously softening. Yield, about 88%. In conc. H.SO, the color is pale blue, it soon darkens and changes to reddish violet when warmed. 3,4,4'-Trimethoxydiphenylacetic acid, MeOCaHaCH-(CO<sub>2</sub>H)C<sub>2</sub>H<sub>2</sub>(OMe), from the preceding compd.; aggregates of flat prisms from AcOH (50%), m. 154-5°. Yield, 82%. In conc. H<sub>2</sub>SO<sub>4</sub> the color is reddish violet, changing to dark red when heated; CO is evolved at about 50° and ceases 160°, the quantity is about 66% of 1 mol. Ammonium salt, microscopic plates from aq. NH.Cl. o-Methoxymandelonitrile is deposited in crystals of the rhombic system, a:b:co.862: 1: 0.462. The following derivs, were prepared in the manner described above: 2-Hydroxy-5-methyl-2'-methoxydiphenylacetolactone (I), from p-cresol; flat prisms, or rectangular plates from glacial AcOH, softens 134°, m. 136°. In conc. H.SO, the color is reddish. Yield, 55%. 2-Hydroxy-5-methyl-2'-methoxydiphenylacetic acid, from the lactone; crystallin crusts with 1 H<sub>2</sub>O from C<sub>2</sub>H<sub>4</sub>, m. 150°. Basic barium salt (III), from the lactone and aq. Ba(OH); light yellow plates, darkens 120°. 2-Hydroxy-5-methyl-2'-methoxydiphenylacetamide, from the lactone and alc. NH, at 100° colorless, microscopic prisms from alc. + H<sub>2</sub>O, decomp. 177-8°. Anilids, colorless prisms from alc. + H<sub>2</sub>O, m. 192-4°, previously softening. Yield, 90%. Hydraside,

granular crystals consisting of microscopic, hexagonal prisms, m. 154°. In conc. H.SO. it slowly develops a blue color, changing to greenish brown on adding a trace of KNO. Bensylidinehydraside, crystals from alc., m. 202°. In conc. H.SO. the color is pale yellow. 3-Methyl-4-hydroxy-2'-methoxydiphenylacetamide, MeOCaHaCH (CONH.)C.H.MeOH, from o-methoxymandelonitrile, o-cresol and H.SO. (73%); hexagonal prisms with 1 AcOH from glacial AcOH, m. 130-1°, previously softening. Yield, 25% of the nitrile. a-Hydroxy-3-methyl-2'-methoxydiphenylacetic lactone (I), is formed together with the preceding compd. and is separated by its insolubility in cold, dil., aq. KOH; granular crystals from toluene, needles from glacial AcOH, m. 164°, previously softening. No pure condensation products could be obtained from 4-Hydroxy-2'-methoxydiphenylacetamide, o-methoxymandelonitrile and m-cresol. MeOC,H,CH(CONH,)C,H,OH, from PhOH, o-methoxymandelonitrile and H,SO. (73%); bundles of colorless, microscopic prisms from glacial AcOH + H<sub>2</sub>O, m. 204-5°. Yield, 40%. It gives no color with conc. H,SO, and is hydrolyzed very slowly by b., conc., aq. KOH. 2-Hydroxy-2'-methoxydiphenylacetic lactone (IV), is formed together

$$MeOC^{4}H^{4}CH < C^{2}H^{4} > O$$

with the preceding compd. and is sepd. by its insolubility in aq. Ba(OH)<sub>1</sub>; prisms from alc. + H<sub>2</sub>O, m. 160-2°, previously softening. Yield, 8%. Anilide, MeOC<sub>4</sub>H<sub>4</sub>CH<sub>4</sub>-(CONHPh)C<sub>5</sub>H<sub>4</sub>OH, from (IV) and PhNH<sub>2</sub>; crystallin crusts from toluene. m. 167°. The statement of Bistrzycki and Simonis (Ber., 31, 2812 (1898)) that mandelonitrile and PhOH give a mixture of o-hydroxydiphenylacetolactone (39-43%) and p-hydroxydiphenylacetic acid (15%), is confirmed. The lactone is separated and purified readily, but the acid is contaminated with amide, consequently the acid is best prepared from mandelic acid and not from the nitrile.

J. BISHOP TINGLE.

Synthesis of Naphthophenazine Derivatives. F. KEHRMANN AND JOSÉ RIERA Y PUNTI. Chem. Sch., Mülhausen i/E. Ber., 44, 2618-21.—2,4-Diaminona phthophenazine, formula (I) below, is prepared by b. a mixture of picryl-\(\beta\)-naphthylamine, alc., conc. H<sub>2</sub>SO<sub>4</sub> and SnCl<sub>2</sub> and is purified by conversion into the Ac deriv.; red, lustrous leaflets from PhNO<sub>2</sub>, sublimes and in part decomp. about 320°. Yield, 20-30% of the nitro compd. In conc. H<sub>2</sub>SO<sub>4</sub> the color is brownish orange-red, changing on the gradual addition of H<sub>2</sub>O to fuchsin-red, then to dull green. Monosuljate, dark green. Diacetyl derivative, from (I), Ac<sub>2</sub>O and anhydrous AcONa; large, golden lustrous,

straw-yellow plates from diphenylamine. m. about 340° (decomp.). In conc.  $H_2SO_4$  the color is dull reddish violet.  $I_1,3$ -Diaminonaphthophenasine (II), is prepared like (I) from picryl- $\alpha$ -naphthylamine; dark red, granular crystals from xylene, sublimes and decomp. 290–300°. In conc.  $H_2SO_4$  the color is brownish red, changing when diluted to Bordeaux-red, then to dull green. Yield, 0.5-1% of the nitro compd. Monosulfate. Diacetyl derivative, small, yellow needles from alc. +  $C_0H_2$  (2:1), m. 320° (decomp.). In alc., or  $C_0H_2$ , the color is yellow, with a green fluorescence; in conc.

H<sub>2</sub>SO<sub>4</sub>, brownish red, when diluted Bordeaux-red. 1,3-Diaminomethylnaphthophenason-ium chloride (III), by heating the preceding compd. with PhNO<sub>2</sub> and Me<sub>2</sub>SO<sub>4</sub>, at 150° and hydrolyzing the resulting compd. with HCl and NaCl; slender, dark red needles from dil., aq. HCl. In H<sub>2</sub>O the color is olive-green in thin layers, dull red in thick ones; in conc. H<sub>2</sub>SO<sub>4</sub>, dull Bordeaux-red, changing when diluted to violet-red, then to olive-brown. Chloroplatinate, (C<sub>17</sub>H<sub>14</sub>N<sub>4</sub>)<sub>2</sub>.H<sub>2</sub>PtCl<sub>6</sub>, small, grayish black crystals.

J. BISHOP TINGLE.

Isomer of Aposafranine and the Third Isomer of Phenosafranine. F. KEHRMANN AND JOSÉ RIERA Y PUNTI. Chem. Schl., Mülhausen i/E. Ber., 44, 2622-7.—Tetranitro-2,4,6,5'-phenylamino-2'-diphenylamine, (O<sub>2</sub>N)<sub>2</sub>C<sub>2</sub>H<sub>2</sub>NHC<sub>2</sub>H<sub>4</sub>(NO)<sub>2</sub>NHPh, is prepared by warming picryl chloride with 4-nitro-2-aminodiphenylamine, AcONa and alc.; brownish yellow crystals from glacial AcOH, darkens and decomp. 174°. In presence of conc., aq. NaOH it condenses to 1,3,7-trinitro-10-phenyldihydrophenazine, formula (I); blackish violet needles from PhNO<sub>2</sub>, decomp. above 265°. In hot, alc.

$$O_{\mathfrak{g}} N \stackrel{\text{NO}_{\mathfrak{g}}}{\longleftarrow} NH - \stackrel{\text{NO}_{\mathfrak{g}}}{\longleftarrow} NO_{\mathfrak{g}} \qquad \qquad N = - \stackrel{\text{NH}_{\mathfrak{g}}}{\longleftarrow} NH_{\mathfrak{g}} \qquad \qquad N = - \stackrel{\text{NH}_{\mathfrak{g}}}{\longleftarrow} NH_{\mathfrak{g}}$$

$$O_{\mathfrak{g}} N \stackrel{\text{NPhCl}}{\longleftarrow} NPhCl = \stackrel{\text$$

NaOH the color is violet; in conc. H<sub>2</sub>SO<sub>4</sub> olive-green, changing to a dark brown ppt. when diluted. It is converted into 2,6-diaminophenylphenazonium chloride (II), by heating with glacial AcOH, SnCl, HCl and subsequent treatment with NaHCO, and NaCl; large, prismatic crystals from highly dil HCl. Its solns. have the following colors: in H<sub>2</sub>O, blue with a violet tinge; in alc., greenish blue, mineral acids change these colors to fuchsin-red; slightly fuming H<sub>2</sub>SO<sub>4</sub>, brown; conc. H<sub>2</sub>SO<sub>4</sub>, greenish blue, changing to fuchsin-red when diluted and to violet-blue when neutralized; dil. HCl red. These results indicate that (II) forms 4 series of salts. Mordanted cotton is dyed dull greenish blue by (II), the color is changed to red by dil. HCl, but is restored by washing. Chloroplatinate, (C18H14N3)2.PtCl4, blackish violet crystals. Monoacetyl derivative, from (II) and AcO; reddish brown needles from aq. NaCl. It dyes mordanted cotton dull violet. In hot H<sub>2</sub>O the color is violet; in conc. H<sub>2</sub>SO<sub>4</sub>, bluish green, changing to reddish violet when mixed with ice. Chloroplatinate, (C<sub>30</sub>H<sub>17</sub>ON<sub>4</sub>)<sub>2</sub>.PtCl<sub>4</sub>, dark brown cryst. powder. Diacetyl derivative, C12H12N2ClAc2, from the mono-Ac compd., Ac<sub>2</sub>O and anhydrous AcONa; brownish red, cryst. granules with a bronze luster. In H<sub>2</sub>O the color is ponceau-red; in conc. H<sub>2</sub>SO<sub>4</sub>, thin layers are olive-green, thick layers purple-red, ice changes this to ponceau-red; in fuming H<sub>2</sub>SO<sub>4</sub>, light brownish red. It dyes mordanted cotton dull violet-red. Chloroplatinate, (C<sub>22</sub>H<sub>10</sub>O<sub>2</sub>N<sub>4</sub>)<sub>2</sub>,-PtCla, light brownish red, cryst. ppt. with a brassy luster. Isoaposafranine chloride (III), is prepared by diazotizing the above mono-Ac deriv. with dil H<sub>2</sub>SO<sub>4</sub> + NaNO<sub>2</sub>, treating the product with alc., subsequently hydrolyzing with dil H.SO, and adding NaCl; black, highly unstable, cryst. powder. Chloroplatinate, (C12H14N2)2.PtCle cryst. powder; in H<sub>2</sub>O the color is violet; in conc. H<sub>2</sub>SO<sub>4</sub>, olive-green, H<sub>2</sub>O changes it to green and it becomes violet when neutralized. The acetyl compd., before hydrolysis, gives the following derivs.: ferric chloride, brown ppt.; nitrate, compact, brownish black crystals from MeOH, when powdered the color is reddish brown, in H<sub>2</sub>O yellow brownish red; in aq. NaOH, fugitive Bordeaux-red. Chloroplatinate, (CmH16ON), PtCla, brownish red crystals. I. BISHOP TINGLE.

Third Isomer of Aposafranine. F. KEHRMANN AND A. MASSLENIKOV Org. Lab., Univ. Lausanne. Ber., 44, 2628-31.—2-Acetamino-3-aminophènyiphenasonium chloride, formula (I) below, is prepared by the interaction of isophenosafranine chloride

(Ber., 33, 3074 (1900)), AcOH and Ac<sub>2</sub>O, at the ordinary temp., during several days; brownish red plates with a bronze luster from H<sub>2</sub>O. In dil. alc. the color is violet-red;

in conc. H<sub>2</sub>SO<sub>4</sub>, greenish yellow, on adding ice it changes to yellowish red, then to violet-red. Chloroplatinate, (C<sub>20</sub>H<sub>17</sub>N<sub>4</sub>O)<sub>2</sub>.PtCl<sub>4</sub>, dark violet, cryst. powder with a bronze luster. Dichromate, resembles the chloroplatinate. Chlorogurate, crystallin powder. Iodide, dark violet, microcrystallin powder. By means of the diazo reaction, in presence of alc., at o°, and subsequent hydrolysis at the ordinary temp., (I) is converted into 1-aminophenylphenazonium chloride (II), which was purified by isolation first as the nitrate, then as the bromide; greenish blue. Nitrate, from NaNOs; yellowish green crystals. Bromide, dark green, granular, lustrous crystals from aq. NaBr. In H<sub>2</sub>O the color is greenish blue; in alc., bluish green; in conc. H<sub>2</sub>SO<sub>4</sub>, greenish yellow, changing successively, on the addition of ice, to reddish yellow, green and blue. Chloroplatinate, (C18H14N2)2.PtCl, green cryst. powder. Dichromate, iodide and chloroaurate, dark green and cryst. Acetyl derivative of the bromide from (II), Ac.O and ZnCl., at the ordinary temp., followed by NaBr; small, brown crystals. In H<sub>2</sub>O the color is Bordeaux-red. In conc. H,SO4, greenish yellow in thin layers, in thicker ones, brownish red, the addition of ice changes the color to orange-red, then to wine-red. Chloroplatinate, (C12H12N2Ac)2.PtCl2, dark brownish red, cryst. powder. J. B. TINGLE.

Two Forms of o-Quinone. Reply. F. KEHRMANN. Org. Lab., Univ. Lausanne. Ber., 44, 2632-3.—Willstätter and F. Müller (C. A., 3, 3079; 5, 3433) regard the color-less, unstable modification of o-quinone as a peroxide, formula (I) below, whereas

$$(I) \qquad (II) \qquad (III) \qquad (IV)$$

$$OH \qquad (OH)$$

$$OH \qquad (OH)$$

the colored modification is supposed to be a true quinone (II). The author points out that the colorless form has never been analyzed and that the presence of Na<sub>2</sub>SO<sub>4</sub> is not sufficient to prove that the compd. may not be a hydrate (III), or (IV). It might even be an additive product of (II) and Et<sub>2</sub>O because, contrary to Willstätter's assumption, such a compd. would not necessarily be more deeply colored than (II).

J. BISHOP TINGLE.

Methylation of the Alcoholic Hydroxyl in Morphine, Codeine and in the Methylmorphimethines. R. PSCHORR AND F. DICKHAUSER. Chem. Inst., Univ. Berlin. Ber., 44, 2633-40.—The methyl derivs. described below were prepared by dissolving the OH compd. in 1 N aq. NaOH (2.5 mol.) and shaking with Me<sub>2</sub>SO<sub>4</sub>, or MeI. Methylcodeine methiodide,  $C_{16}H_{14}O(OMe)_2NMe_2I$ , is prepared from morphine, or codeine; rods with 2 H<sub>2</sub>O from alc. (80%), decomp. 257° (cor.); in H<sub>2</sub>O,  $[\alpha]_D^{22} = -107.2^\circ$ . Yield, about 90%.  $\alpha$ -Dimethylmorphimethine,  $C_{16}H_{12}O(OMe)_2NMe_2$ , is obtained by the action of hot, aq. NaOH on the preceding compd.; lustrous plates, or broad needles, from MeOH + H<sub>2</sub>O, m. 94°; in MeOH,  $[\alpha]_D^{24} = -251.9^\circ$ . Yield, about 85%. Methiodide, flat needles from H<sub>2</sub>O, decomp. 256° (cor.); in H<sub>2</sub>O,  $[\alpha]_D^{23} = -134.4^\circ$ .  $\beta$ -Dimethylmorphimethine is prepared by b. the  $\alpha$ -compd. with aq., alc. KOH, or Ac<sub>2</sub>O, or with Ac<sub>2</sub>O at 210°; oil. Methiodide, prismatic needles from H<sub>2</sub>O, decomp. 318-20° (cor.); in H<sub>2</sub>O,  $[\alpha]_D^{22} = 268.5-78.5^\circ$ . Yield, about 80 and 60% from the KOH and AcOH, resp. The

methylmorphimethines when methylated as described above, give  $\alpha$ - and  $\beta$ -dimethylmorphimethine methiodides, which are identical with those just described.  $\gamma$ -Dimethylmorphimethine methiodide, lustrous plates from H<sub>2</sub>O, or alc., decomp. 259° (cor.); in H<sub>2</sub>O [ $\alpha$ ]<sup>20</sup> 14°.  $\delta$ -Dimethylmorphimethine methiodide, slender needles from H<sub>2</sub>O, prisms from alc., decomp. 286° (cor.); in H<sub>2</sub>O, [ $\alpha$ ]<sup>20</sup> 170.9°.  $\epsilon$ -Dimethylmorphimethine methiodide, long needles from H<sub>2</sub>O, long plates from alc., decomp. 277° (cor.); in MeOH, [ $\alpha$ ]<sup>20</sup> —79.4°. At 165°, aq., alc. KOH converts  $\beta$ -dimethylmorphimethine methiodide into morphenol, which was identified as the Ac deriv. Yield, about 70%. When methylated in the manner described above, benzyl alc. gives benzyl methyl ether (yield about 75%) and cinnamyl alc. gives cinnamyl methyl ether (yield about 50%). The method was not advantageous with amyl or octyl alcs., nor with borneol, or isoborneol. The benzyl methyl ether is most conveniently purified and separated as the hydroferrocyanic acid deriv.

J. BISHOP TINGLE.

Tetraalkyl Silicanes. ARTUR BYGDEN. Chem. Lab., Univ. Upsala. Ber., 44, 2640-52.—The alkyl silicanes were prepared by Kipping's method, from SiCl, and alkyl Mg haloid, in abs. Et,O, various proportions of the RMgX being employed. Tetramethylsilicane, SiMe, b7014 26-7°; mol. wt., 89.7 (V. Meyer). Trimethylethylsilicane, EtSiMea, from MeMgBr and EtSiCla; colorless liquid, b716-4 62.6-3.4° (cor.); mol. wt. 103.5 (V. Meyer). Yield, about 28% of the EtSiCl2; with MeMgI the yield is only about 8%. Trimethylpropylsilicane, from MerSiPra, from PraSiCla and MeMgBr; liquid b<sub>750-8</sub> 89.3-6° (cor.). Yield, of pure compd., 27% of the chloride. Dimethyldiethylsilicane, Et.SiMe, from Et.SiCl, and MeMgI; liquid, b75918 95.5-6.0° (cor.). Yield, about 24% of the chloride. Butylsilicon trichloride, BuSiCla, was obtained from BuMgBr and SiCl, but could not be purified completely; b. 148.5-9.5°. With MeMgBr it gives trimethylbutylsilicane, MezSiBu; liquid, b732.9 115.2° (cor.). Yield of pure compd., 37% of the chloride. Isobutylsilicon trichloride was prepared like the isomer, but could not be purified completely; liquid, fumes in air, b. 138-43°; d4 1.161. It was converted into trimethylisobutylsilicane, but the product could not be purified completely; b<sub>784</sub> 107.9-8.2°. Ethylpropylsilicon dichloride, EtSiPraCl<sub>2</sub>, was prepared with difficulty from PrSiCl, and EtMgBr; colorless liquid, fumes in air, b. 152-8°; d4 1.048. With MeMgBr it gives dimethylethylpropylsilicane, Me SiEtPra; liquid, b<sub>700-4</sub> 120-2° (cor.). Yield, about 65% of the chloride. Trimethylisoamylsilicane, Me<sub>2</sub>SiC<sub>2</sub>H<sub>11</sub>, from isoamylsilicone trichloride and MeMgBr; liquid, b<sub>786-4</sub> 131-2° (cor.). Yield, 63.3% of the chloride. Ethylisobutylsilicone dichloride, EtSiBu<sup>B</sup>Cl<sub>2</sub>, is best obtained from isobutylsilicone trichloride and EtMgBr; liquid, b. 168-70°; d45 1.028. It was not purified completely; the yield is larger by this method than from EtSiCl. and BusMgBr. With MeMgBr it gives dimethylethylisobutylsilicane, Me,SiEtBus, liquid, b<sub>700</sub> 137-9° (cor.). Yield, about 25% of the chloride. Tetraethylsilicane was obtained in good yield from SiCl<sub>4</sub> and EtMgBr; b<sub>784.6</sub> 152.8-3.2° (cor.); do° 0.7802. Dimethylpropylsilicane could not be purified. The following additional physical constants are given, the numbers in Roman type refer to the  $d_4^{16}$ , those in italics to the sp. vol. and those in parentheses to the mol. vol., both at 15°: Me,Si, 0.6510, 1.5361, (135.7); Me<sub>3</sub>SiEt, 0.6901, 1.4491, (148.3); Me<sub>3</sub>SiPr<sup>a</sup>, 0.7063, 1.4158, (164.7); Me\_SiEt, 0.7214, 1.3862, (161.3); Me\_SiBua, 0.7227, 1.3837, (180.4); Me\_SiEtPra, 0.7347, 1.3611, (177.4); Me,SiBu<sup>\$\beta\$</sup>, 0.7322, 1.3657 (197.2); Me,SiEtBu<sup>\$\beta\$</sup>, 0.7463, 1.3399, (193.5); Et<sub>4</sub>Si, 0.7694, 1.3997, (187.7). J. BISHOP TINGLE.

"Lakes." [Ketostannic Chloride Derivatives.] I. P. PFEIFFER Chem. Lab., Univ. Zurich. Ber., 44, 2653-62.—Unless otherwize stated the Sn compds. described below were prepared from the resp. ketones and SnCl., in b., anhydrous C.H., only the name of the ketone is mentioned. o-Hydroxyacetophenone, AcC.H.OSnCl., pale yellow crystals, m. about 238°. It is decomp. by b. H2O. o-Hydroxybensophenone, BzC.H4-

OSnCl<sub>2</sub>, light yellow, lustrous plates, m. about 250°. Resacetophenone, HOC<sub>2</sub>H<sub>2</sub>AcO-SnCl<sub>2</sub>, compact, light yellow, transparent, lustrous crystals, m. about 235-48°. Quinacetophenone, lustrous, deep yellow, prismatic crystals. Euxanthone,

small orange-yellow crystals. Gallacetophenone, HOC<sub>2</sub>H<sub>2</sub>AcO<sub>2</sub>SnCl<sub>2</sub>, deep yellow powder, decomp. without m. In pyridine the color is orange-yellow, in alc. brownish yellow. Gallobenzophenone, HOC<sub>2</sub>H<sub>2</sub>BzO<sub>2</sub>SnCl<sub>2</sub>, orange-colored powder; when heated it becomes red then black and tarry. The constitution of the above compds. is discussed at great length.

J. BISHOP TINGLE.

Constitution of the Pseudobases of Quinoline. II. ADOLF KAUFMANN AND J. M. Pla y Janini. Univ. Lab., Geneva Ber., 44, 2670-7; cf. C. A., 5, 1908.-2-Phenylquinoline methiodide is best prepared from PhMgBr and quinoline methiodide, the resulting product being immediately oxidized with alc. I, in presence of AcONa. Yield, 90-5%. In H<sub>2</sub>O the soln. is colorless; in alc., yellow; in CHCl<sub>2</sub>, orangered. Aq. NaOH hydrolyzes it to the pseudobase o-methylaminocinnamyl phenyl ketone, MeNHC<sub>e</sub>H<sub>4</sub>CH: CHBz; flocculent and unstable. With HCl it regenerates 2-phenylquinoline methochloride, which was also prepared from the methiodide and AgCl; long, almost colorless needles from alc. + Et,O, decomp. about 170°, m. 195-200°. In H<sub>2</sub>O, with KOH and PhNH<sub>2</sub>, either of the above quaternary salts forms the anil, MeNHC, HaCH: CHCPh: NPh; greenish yellow needles from ligroin, m. 140°. It is hydrolyzed readily by dil. mineral acids. In presence of alkali, air, or more rapidly K<sub>2</sub>Fe(CN), oxidizes the pseudobase to a compd., probably benzoyl-omethylaminoacetophenone, MeNHCaHaCOCHaBz; opaque needles from CaHa, m. 123°. It is very stable. Chloride, C16H14NOCl, almost colorless, stellate needles, m. 237° (decomp.). Iodide, pale yellow, m. 220°. Picrate, yellow crystals from alc.,

m. 180°. These salts are supposed to have the cyclic formula, C.H. NMeX

At 250-60° the chloride decomp. into MeCl and 2-phenyl-4-oxyquinoline. Distillation with Zn dust reduces it to 2-phenylquinoline.

J. BISHOP TINGLE.

Phthaleins of 3,5,3',5'-Biresorcinol. RICHARD MEYER AND KARL MEYER. Chem. Lab., Techn. Hochsch., Braunschweig. Ber., 44, 2678-84.—Benedikt and Julius' "soluble phthalein" (Monatch., 5, 180 (1884)) from biresorcinol, phthalic anhydride and conc. H<sub>2</sub>SO<sub>4</sub> darkens on exposure to light, decomp. in contact with acid vapors and gives a deep blue color with alkali; mol. wt., in b. acetone, 322-8. Tetraacetate, from Ac<sub>2</sub>O + conc. H<sub>2</sub>SO<sub>4</sub>; large, flat, rhombic crystals from glacial AcOH + H<sub>2</sub>O, m. 237-8°. Tetrabenzoate, from BzCl and alkali; thick, prismatic needles from glacial AcOH, m. 180-1°. Tetramethyl ether, from Me<sub>2</sub>SO<sub>4</sub> and aq. NaOH; colorless needles from glacial AcOH, does not m. Tetraethyl ether, from Et, SO, and aq. NaOH, or EtI and alc. EtONa; flat needles, or plates from alc., m. 223°. Tribromo derivative, C<sub>20</sub>H<sub>2</sub>O<sub>4</sub>Br<sub>2</sub>, rrom Br in glacial AcOH; slender needles from glacial AcOH. The "insoluble phthalein" is formed together with the above "sol." compd., but the yield is increased if the anhydride and phenol are taken in the ratio 1: 2 mols. Hexabenzoate, from BzCl and alkali; broad plates from CHCl, and Et,O, m. 245-50°. Hexaethyl ether, from Et, SO, and alkali; needles from CHCl, + Et, O, m. 234-6°. Bisbenzeneazobiresorcinol, PhN: NC<sub>2</sub>H<sub>2</sub>(OH)<sub>2</sub>C<sub>2</sub>H<sub>2</sub>(OH)<sub>2</sub>N: NPh, is formed by adding biresorcinol, in alkali, to a phenyldiazonium salt, at oo; thick, reddish brown needles with a steel-blue luster from glacial AcOH. Bis-p-tolueneazobiresorcinol, MeC,H4N: NC,H2(OH),C,H2(OH)<sub>2</sub>N: NC<sub>2</sub>H<sub>4</sub>Me, prepared like the preceding compd., which it resembles closely; slender needles from glacial AcOH. These results show that the "sol." and "insol." phthaleins have formulas (I) and (II), resp.

J. B. TINGLE.

Walden's Inversion. VII. Optically Active Leucinic Acid ( $\alpha$ -Hydroxyisohexoic Acid) and its Conversion into a-Bromoisohexoic Acid. Helmuth Scheibler and ALVIN S. WHEELER. Chem. Inst., Univ. Berlin. Ber., 44, 2684-90; cf. C. A., 4, 2822; 5, 3282.—dl-α-Hydroxyisohexoic acid (dl-leucinic acid) is best prepared from I N aq. NaOH and com.  $\alpha$ -bromoisohexoyl bromide; rhombic plates from anhydrous Et<sub>2</sub>O + petroleum ether, m. 76-7°. Yield, 78%. It is resolved into its optically active components by the help of brucine, quinine, or quinidine, of which the latter is the more convenient. In each case the salt of the l-acid crystallizes first. l-Acid, thin prisms from Et<sub>2</sub>O + petroleum ether, softens about 78°, m. 81-2°; in 1 N aq. NaOH,  $[\alpha]_{D}^{20}$  -27.8° (±0.2°); in H<sub>2</sub>O,  $[\alpha]_{D}^{20}$  -10.4° (±0.2°). The d-acid was not completely purified. The l-acid was also prepared by diazotizing l-leucine. Barium salt, crystals from H<sub>2</sub>O. The d-acid was prepared in a similar manner from d-leucine; m. 80°; in 1 N aq. NaOH,  $[\alpha]_D^{20}$  26.3° (±0.2°). Ethyl dl- $\alpha$ -hydroxyisohexoate, is obtained by b. the dl-acid with alc. containing 1.5% of HCl; liquid with a faint, pleasant odor, b<sub>16</sub> 80-1°. Yield, 80%. Ethyl l-a-hydroxyisohexoate is prepared like the preceding compd.;  $b_{12}$  79-80°;  $[\alpha]_{\mathbf{p}}^{20}$  —11.07° (±0.02°); the value for the pure compd. is probably -11.44°. At the temp. of a freezing mixture, red P and Br convert the dl- and l-esters into the a-bromoisohexoic esters. The l-compd. was, in part, transformed into the dl-ester. Ethyl dl-a-bromoisohexoate, liquid, b<sub>11</sub> 86-7°. Yield, 36%. These results and those obtained previously show the following relationship of l-leucine to the Walden inversion: l-leucine  $\frac{HNO_2}{l}$  l- $\alpha$ -hydroxyisohexoic acid  $\longrightarrow l$ -ester  $\frac{Br+P}{l}$ d- $\alpha$ -bromoisohexoic ester. l-Leucine  $\longrightarrow$  l-ester NOBr d- $\alpha$ -bromoisohexoic ester. l-Leucine NOBr l- $\alpha$ -bromoisohexoic acid  $\longrightarrow$  l-ester. J. BISHOP TINGLE.

Carbon Double Linkage. III. Bromides of Anisalcinnamalacetone. H. BAUER AND H. DIETERLE. Lab. Pure and Pharm. Chem., Roy. Techn. Hochsch., Stuttgart. Ber., 44, 2691-7.—Anisalcinnamalacetone, PhCH: CHCH: CHCOCH: CHC<sub>4</sub>H<sub>4</sub>OMe, was prepared from anisic aldehyde and cinnamalacetone (yield, about 80%), or from anisalacetone and cinnamic aldehyde, by the action, in each case, of warm, alc., aq. KOH; yellow, lustrous plates from alc., or CS<sub>2</sub>, m. 138-9°. In conc. HCl the color is dark carmoisin-red, in conc. H<sub>2</sub>SO<sub>4</sub>, yellow. Dibromide, PhCH: CHCH: CHCO-CHBrCHBrC<sub>2</sub>H<sub>4</sub>OMe, was formed in glacial AcOH + Et<sub>2</sub>O soln.; small, slender, color-less needles from CS<sub>2</sub>, m. 139-40°. Tetrabromide, PhCH: CHCHBrCHBrCOCHBr-CHBrC<sub>4</sub>H<sub>4</sub>OMe, prepared like the dibromide, but from 4Br; slender, colorless needles, m. 155-6°. 1-Phenyl-7-anisyl-6-bromo-7-methoxy-5-heptadieneone, PhCH: CHCH: CHCOCHBrCH(OMe)C<sub>4</sub>H<sub>4</sub>OMe, is formed by b. the dibromide with MeOH, during 10 mins.; light yellow plates from MeOH. In conc. HCl the color is yellow; in conc.

H<sub>2</sub>SO<sub>4</sub>, deep bluish violet. In acetone, KMnO<sub>4</sub> oxidizes it to anisic aldehyde and γ-phenyl-α,β,γ-trihydroxybutyrolactone. In alc., at the ordinary temp., Mg(OAc)<sub>8</sub> and the above dibromide give *1-phenyl-γ-anisyl-6-bromo-γ-acetoxy-5-heptadieneone*, PhCH: CHCH: CHCOCHBrCH(OAc)C<sub>8</sub>H<sub>4</sub>OMe; opaque, pale yellow prisms from alc., m. 78-9°. *1-Phenyl-γ-anisyl-6-bromo-γ-methoxy-5-heptadieneone* 3,4-dibromide, PhCH: CHCHBrCHBrCOCHBrCH(OMe)C<sub>8</sub>H<sub>4</sub>OMe, from the methoxy compd. and Br, in glacial AcOH, at 0°, or from MeOH and the above tetrabromide; slender, white needles from CS<sub>2</sub>, m. 176-7.5°. It is not changed by b. MeOH, but b. pyridine converts it into the dibromo compound, PhCH: CHCBr: CHCOCHBrCH(OMe)C<sub>8</sub>H<sub>4</sub>OMe; slender, yellowish brown plates, m. 174-5°. It is oxidized by KMnO<sub>4</sub> to BzOH and anisic acid.

Action of Ammonia on Unsaturated Acids. II. G. L. STADNIKOV. Org. Lab., Univ. Moscow. J. Russ. Phys. Chem. Soc., 42, 885-90.—In order to show that imino-dicarboxylic acids are formed by the action of primarily produced amino acids on unsatd. acids (cf. Ibid., 41, 900), the NH<sub>4</sub> salt of glycocoll was made to react upon the NH<sub>4</sub> salt of crotonic acid, the resulting product esterified and fractionated. Fraction b<sub>16</sub> 62-8° consisted of ethyl β-aminobutyrate; fraction b<sub>16</sub> 144° was diethyliminobutyrate-Me.CH.CH<sub>2</sub>CO<sub>2</sub>Et

acetate, ; d<sub>2</sub><sup>20</sup> 1.034; n<sub>D</sub><sup>20</sup> 1.437. By saponifying this ester the free NH—CH<sub>2</sub>CO<sub>2</sub>Et

acid was obtained with slight decomp. The yield of ester can be increased to 45% by using the Na instead of the NH<sub>4</sub> salts. By action of EtCHO and KCN on glycocoll-6-ethyliminodiacetate hydrochloride, α-iminoacetic-butyric acid, C<sub>4</sub>H<sub>11</sub>NO<sub>4</sub>.H<sub>2</sub>O, was obtained; m. 104-5°. By hydrolyzing the Et ester with HCl its hydrochloride, C<sub>4</sub>H<sub>11</sub>-NO<sub>4</sub>.HCl, was obtained; m. 175-7° H. M. GORDIN.

Cyclobutylcarbinol and its Isomerization into Derivatives of Cyclopentane by Acids. N. YA. DEMYANOV. Chem. Lab., Agric. Inst., Moscow J. Russ. Phys. Chem. Soc., 42, 837-55.—By heating the Et tetramethylenecarboxylate with Na in abs. alc. to 140-50° a yield of 51% of cyclobutylcarbinol (I) (cf. Perkin, J. Chem. Soc., 79, 329) was obtained, b<sub>780</sub> 140-2.5°, d<sub>10</sub> 0.9199, d<sub>20</sub> 0.9129, n<sub>20</sub> 1.4449. Upon oxidation (I) is changed

CH<sub>2</sub>.CH.CO<sub>3</sub>.CH<sub>2</sub>.CH.CH<sub>3</sub>, to the cyclobutylcarbinyl tetramethylenecarboxylate, | | b. 218-CH<sub>2</sub>.CH<sub>3</sub>.CH<sub>4</sub>.CH<sub>4</sub>

20°;  $d_{10}^{10}$  0.9795;  $n_{10}^{10}$  1.4546;  $n_{10}^{21}$  1.4533. By the action of HBr on (I) at 100° bromocyclopentane,  $C_aH_aBr$ , was obtained, b. 137-9°;  $d_{10}^{10}$  1.385;  $n_{10}^{10}$  1.4875. That the bromide is an isomerized cyclopentane deriv. and not a deriv. of methylcyclobutanol, was shown by the constants of the hydrocarbon obtained by reducing it with Zn + Pd in HBr soln., since general considerations would require methylcyclobutane to have lower constants than cyclopentane. Iodocyclopentane was obtained (assisted by Pitman) by a method similar to that for the bromide;  $b_{17}$ -as 80-3°;  $b_{70}$ 0 162-4°;  $d_{1}^{0}$ 1.1754;  $d_{1}^{21.5}$ 1.6825;  $n_{10}^{22}$ 1.5374. Attempts to change the iodide into the corresponding nitro compd. (by means of AgNO<sub>2</sub>) were not successful; instead cyclopentane pseudonitrol,  $C_bH_a(NO_2)(NO)$ , was obtained. On heating (I) with  $(CO_2H)_2$  to 170-80° (assisted by

CH<sub>2</sub>.CH CH<sub>2</sub>.C : CH<sub>3</sub>.

DOYARENEO) a mixt. of 2 hydrocarbons, | CH<sub>2</sub>.CH and | was formed, CH<sub>3</sub>.CH<sub>4</sub>.

 $b_{783}$  44.2-44.6°;  $d_{4}^{0}$  0.792;  $d_{4}^{18}$  0.774;  $n_{2}^{18}$  1.4217. Yield, 60%. On digesting this mixt. with  $H_{2}$ SO<sub>4</sub> it is partly transformed into *cyclopentanol* ( $b_{783}$  137-8°) oxidizable to cyclopentanone.

Determining Halogens in Organic Compounds (WARUNIS). 7.

Preparation of Neutral Ammonium Citrate Solutions (HALL). 7.

Velocity of Hydrolysis of Glycerophosphoric acid (MALENGREAU, PRIGENT). II. Precipitation with Phosphotungstic Acid (WECHSLER). 11.

Essence of Bubleurum fruticosum Linn (Francesconi, Sanna). 17.

Action of Nitrosyl Chloride on Bubleurum fruticosum (Francesconi, Serna-GIOTTO). 17.

Simultaneous Determination of Elements of an Organic Compound (AUZIES). 7.

Electrical Conductivity of some Organic Compounds (SAKHANOV). 2.

Elaidin Reaction (FOKIN). 27.

Color and Constitution (PORAI-KOSHITZ). 2.

Photokinetics of Bromine Substitution (BRUNER, CZARNECEI). 2.

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## 11. BIOLOGICAL CHEMISTRY.

WM. J. GIES.

GENERAL.

The Enzymes of Diastase. L Lyalin. J. Russ. Phys. Chem. Soc., 42, 624-33. -Pure diastase was prepared by pptn. with (NH<sub>4</sub>)<sub>x</sub>SO<sub>4</sub>, and its action compared with the action of samples on the market. The latter were in every respect less active. It was also shown that the saccharifying effect of diastase on starch corresponds with its solvent properties, and that in drying malt loses some of its fermentative power. The proteolytic and coagulating power of diastase and the oxidizing power of oxidase run parallel with the saccharifying and solvent properties. H. M. GORDIN.

Biochemical Classification of the Proteins. Jose R. CARRACIDO. Anales soc. espan. fis. quim., 8, 261-4.—The following is a biochem. classification of the proteins following the order of succession in the course of biological processes and is a revision of a scheme previously published (Rev. Ibero-Americana cien. med., 7, 87): Albuminoids formed by progressive evolution. A. PROTAMINES. B. PROTEINS. I. Histones. II. Albumins. III. Globulins. IV. Fibrins. C. PROTRIDS. I. Glucoproteids. II. Phosphoproteids. 1. Phosphoproteins. 2. Lecithoproteins 3. Histoproteids a. Cytoproteids. (1) Cyteins. (2) Proteocyteins. b. Nucleoproteids. (1) Nucleins: (a) Nucleoprotamines, (b) nucleohistones, (c) nucleoglobulins. (2) Proteonucleins. (3) Glucoproteonucleins. III. Chromoproteids. Albuminoids formed by retrogressive melamorphosis. A. Albumoids. I. Collagens. II. Keratins. B. Polypeptides by hydrolysis. I. Acidalbumins. II. Alkalialbumins. III. Albumoses. IV. Peptones. H. S. Paine.

The Hemolytic Action of Rice Fat (from Oryza sativa L.). Study of Hemolysis by Fatty Acids. J. Shimazono. Pharm. Inst., Univ. Tokio. Arch. exp. Path. Pharm., 65, 361-6.—The alc. and ethereal extracts of rice act hemolytically. The active substance was isolated and identified as palmitic acid. The lower members of the fatty acid series up to caproic acid, when tested as Na salts, were inactive. Nonylic acid forms a transition member being weakly hemolytic. The acids higher than capric showed an activity not less than oleic. With the very high acids the insolubility of the Na soaps was a disturbing feature.

Walter A. Jacobs.

Solubility of Pancreatic Lipase. L. BERCZELLER. Physiol. chem. lab., Univ. Budapest. Biochem. Z., 34, 170-5.—Lipase is not taken up by ether, olive oil or oleic acid. Its action is not inhibited by shaking. Fat splitting by pancreatic lipase is a heterogenetic reaction. Lipase is not dissolved in any of the phases; it is always present in the form of a suspension.

G. M. MEYER.

Enzyme Action and Electrolytic Dissociation. Hugo Rohonyi. Physiol. chem. lab., Univ. Budapest. Biochem., Z., 34, 176-91.—Active and inactivated enzyme solns. have the same electrolytic dissociation, provided the water is replaced which evaps. during the process of inactivation. The dissociation increases during starch digestion because adsorbed salt enters into soln. There is no change in dissociation during cane sugar inversion.

G. M. Meyer.

Observations on the Mechanism of the Action of Proteolytic Enzymes. A. Ferneral And M. Schoen. Compt. rend., 153, 133-6.—Proteolysis is considerably influenced by the presence of KH<sub>2</sub>PO<sub>4</sub> and K<sub>2</sub>HPO<sub>4</sub>. Expts. were carried out with papayotin (Merck), pancreatin powder and the proteolytic enzyme of the Tyrothrix tenuis. KH<sub>2</sub>PO<sub>4</sub> favors proteolysis and produces a more profound cleavage of the protein mol. The results were identical with fibrin, egg white, casein, and gelatin. The extent of proteolysis was detd. by the quantity of protein not pptd. by tannin.

G. M. Meyer.

The Specific Action of Various Iron Compounds with Respect to their Peroxidasic Properties. J. Wolffand E. de Storcklin. Compt. rend., 153, 139-41.—The presence of small amts. of alkali thiocyanate greatly increases the catalytic action of Fe salts (nitrate, sulfate, chloride and acetate). This activity increases with addition of thiocyanate to a max. when an equimol. quantity, as represented by the formula (CNS)<sub>8</sub>K<sub>8</sub>Fe, is present. Further addition of KCNS exerts no further action. Certain Fe salts or combinations of such may oxidize 1 phenol and be incapable of oxidizing any other. The action of these mixts. is sp. An inappreciable amt. of ferri-ferrocyanide will oxidize hydroquinone to quinhydrone; ferri-thiocyanate will only accomplish this in the presence of a large excess of KCNS. Under similar conditions pyrogallol is not oxidized to purpurogallin.

G. M. Meyer.

Protoplasmic Permeability. Josef Szües. Pflanzenphysiol. Inst., Univ. Wien. Sitzungsber. Wiener Akad., 119, 737-73; through Zentr. Biochem. Biophys., 11, 869.—Cells of Spirogyra and Lemna take up such aniline dyes as methyl violet B and neutral red hydrochloride with a rapidity proportional to the diffusing dye. Fick's law of diffusion is followed. The permeability of the protoplasmic membrane is not const. Such electrolytes as NaNO<sub>3</sub>, KNO<sub>3</sub>, NaCl, Ca(NO<sub>3</sub>)<sub>2</sub>, Mg(NO<sub>3</sub>)<sub>3</sub>, AlCl<sub>3</sub> and FeCl<sub>4</sub> delay the absorption of the dye, increasingly with the valence of cations. It is dependent on the conc. of the electrolytes. Dil. solns. of the electrolyte are proportionally more active than more conc. solns. The absorption of basic dyes can be hindered, also, by such acid dyes as Congo red. The action of the acid dye appears to be a combination

with the basic dye, forming a salt which is impermeable to the protoplasmic wall. The electrolyte exerts action on the plasma.

I. K. Phelips.

The Effect of Anesthetic Substances on the Osmotic Properties of the Plasma Membrane. W. W. LEPESCHKIN. Ber. bot. Ges., 29, 349-55.—From the former work of the author (C. A., 5, 3298) it was to be expected that such substances which are not easily sol. in anesthetics but sol. in H<sub>2</sub>O would pass through the plasma membrane more slowly during anesthesia than normally. Methylene blue and methyl green are sol. in aq. CHCl, and insol. in ether. Thus, using Spirogyra, it was noticed that less of these stains accumulated in the anesthesized cells than in the normal cells in a given time, while Bismarck brown, which is somewhat more sol. in ether than in H<sub>2</sub>O, showed no difference. Furthermore, plasmolysis expts. with KNO<sub>2</sub> and cane sugar showed for normal cells an isotonic coeff., K = 2.95 and 2.98 and a permeability coeff.,  $\mu = 0.127$  and 0.119; for cells anesthesized with CHCl<sub>2</sub>, K = 3.04,  $\mu$  0.100, anesthesized with ether K = 3.12,  $\mu = 0.076$ . These expts. are contradictory to the mosaic structure hypothesis of the plasma membrane of Nahtanson, for they indicate that the way in which H<sub>2</sub>O sol. substances pass through the membrane is identical to that taken by fat sol., or anesthetic substances. The lowering of the permeability of the plasma membrane also produces an increase in the osmotic pressure of the cell sap., amounting to 0.1-0.5 atms. for Spirogyra cells. H. A. SPOEHR.

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## METHODS AND APPARATUS.

Enzyme Studies. II.. The Measurement and Importance of the Hydrogen Ion Concentration in Enzyme Reactions. S. P. L. SÖRENSEN. Compt. rend. trav. lab. Carlsberg, 8, 1-168 (1909); Biochem. Z., 21, 131-304 (1909); cf. C. A., 4, 607.—Having shown that the H+ conc. and not the acidity of a soln. is the important factor in enzyme reactions, S. measured [H+] in such solns. by (1) the electrometric and (2) the colorimetric method. In (1) H is calc. from the e. m. f. of a cell consisting of a HgCl electrode, and a H electrode in the soln. to be tested, with KCl as the connecting soln. From measurements with HCl from 0.1 to 0.01 N, he found  $\pi_0$  for N [H<sup>+</sup>] = 0.3377 V, and from measurements with NaOH soln., he calc.  $K_{W10} = 0.72 \times 10^{-14}$ . [H<sup>+</sup>] was detd. for various mixts. of (a) glycocoll and NaOH or HCl; (b) KH,PO, and Na,HPO. (c) disodium citrate and NaOH or HCl; and (d) Na<sub>2</sub>B<sub>4</sub>O, and NaOH or HCl; the results being shown graphically. The same solns, were studied colorimetrically with a large number of indicators, of which 20 were selected as being least affected by proteins, etc., and therefore best suited for enzyme work. Permanent colors may be added to the standards when colored solns. are to be studied. The importance of H+ in the reaction of invertin, catalase and pepsin is illustrated by examples.

WILLIAM BLUM.

Heynsius' Test Applied to the Detarmination of Albumin in Feces. L. De Jager. Leeuwarden, Holland. Zentr. ges. Physiol. Path. Stoffw., 5, 629-30.—A note in which J. recommends the above test. The feces are extracted, as in Simon's method, with water and the soln. is filtered. A slight excess of AcOH is added and the soln., boiled if a clear filtrate is not readily obtained. Heynsius' test is then applied. This consists in decomposing urin with AcOH and heating to b. The ext. is then divided into 2 portions. To one there is added an equal vol. of satd. NaCl soln.; to the other an equal vol. of water. If albumin is present the NaCl soln shows more turbidity than the other. In doubtful cases, when testing urin for albumin, J. also recommends this test. The NaCl must be added in soln.

C. A. Brautlecht.

Liebermann's Protein Reaction. W. A. VAN EKENSTEIN AND J. J. BLANKSMA. Amsterdam. Chem. Weekblad, 8, 313-5.—The reaction is due to the tryptophan and the furfural radicals contained in protein. It is not given by tyrosine but similar reactions are given by skatole in the presence of sugars, "less-similar" reactions by indole. HCl with tryptophan and protein or skatole when boiled with (1) ω-methoxy-furfural (hexose) gives first red then violet-red, with (2) δ-methylfurfural (methylpentose) gives red to purple-red, with (3) furfural (pentose) gives dirty red to brown: HCl, tryptophan and indole give with (1) light yellow to orange-red, with (2) light yellow to orange-red, with (3) brown-red.

V. E. Henderson.

The Technic of Precipitation with Phosphotungstic Acid. E. WECHSLER. Physiol. Inst. Heidelberg. Z. physiol. Chem., 73, 138-43.—The decomp. of phosphotungstates with Ba(OH)<sub>2</sub> is conveniently effected, even in the cold, by taking advantage of their more or less complete solubility in acetone + H<sub>2</sub>O. The figures give in % the solubility of phosphotungstates (dried over H<sub>2</sub>SO<sub>4</sub>) in a mixt. of 4 vols. acetone and 3 vols. H<sub>2</sub>O; unless otherwise noted they were pptd. from aq. soln. without addition of mineral acid; protoproteose 0.67, the same from acid soln. 6.76, deuteroproteose (acid) 2.94, heteroproteose (acid) 2.24, d- and dl-arginine 120-130, l-histidine 160, d-lysine 140, guanidine 22.8, creatinine 13.34, guanine (acid) 7.21, adenine (acid) 10.50. Fresh, moist phosphotungstates of the proteoses show a greater solubility than the above.

The Velocity of the Hydrolysis of Glycerophosphoric Acid. F. Malengreau AND GEORGES PRIGENT. Biol.-chem. Lab., Univ. Louvain. Z. physiol. Chem., 73, 68-84.—The decomp. of C<sub>2</sub>H<sub>4</sub>(OH)<sub>2</sub>H<sub>2</sub>PO<sub>4</sub> by H<sub>2</sub>O is a case of autocatalysis. There is an optimum acidity above and below which the stability of the compd. increases; this optimum is represented by the acid glycerophosphates. Acids act as negative catalyzers. In spite of this the reaction velocity is not affected by the liberated H<sub>2</sub>PO<sub>4</sub> since in the course of hydrolysis the H+ conc. does not alter. The reaction proceeds therefore as a monomol. one. For the detn. of k o. 1 N solns. were used, and hydrolysis was effected at a temp. of 100°. The H<sub>2</sub>PO<sub>4</sub> liberated was detd. gravimetrically. For  $C_2H_4(OH)_2H_2PO_4$  k = 0.01025; for  $C_2H_4(OH)_2NaHPO_4$  0.02136; for  $C_2H_4(OH)_2$ KHPO<sub>4</sub> 0.02340. For the acid in 1% AcOH k = 0.01005; in 1% HCl, 0.00510; in 1% H<sub>2</sub>SO<sub>4</sub> 0.00621; in 10% H<sub>2</sub>SO<sub>4</sub> 0.00471. Isohydric solns. of HCl and H<sub>2</sub>SO<sub>4</sub> (2.9 and 8% resp.) had identical effects. Salts influence the velocity in accordance with their effect upon the H+ conc. Rise of temp. notably accelerates the reaction; for the acid Na salt k has at 98° the value 0.01657. A. HUNTER.

Quantitative Determination of Volatil Aliphatic Acids. E. EDELSTEIN AND E. WELDE. Z. physiol. Chem., 73, 152-6.—Reply to criticisms of R. S. McCaughey; see C. A., 5, 3080.

A. H.

Malarial Pigment (So-called Melanin): Its Nature and Mode of Production. W. H. Brown. Path. Lab., Univ. of Wisconsin. J. Exp. Med., 13, 290-9.—Two

A. HUNTER.

methods for the study of malarial pigment are described, one for obtaining a solm of pigment from fixed tissues without the removal of hemoglobin from the red blood cells and a method for obtaining an Fe reaction in malarial pigment. By comparing the bleach reactions and solubility of melanins and pigment, the dissimilarity of the 2 classes was demonstrated. Melanin granules are readily bleached by KMnO<sub>4</sub> and H<sub>2</sub>O<sub>3</sub> whereas malarial pigment is unchanged after 48 hrs. treatment with them. Malarial pigment is dissolved by satd. Li<sub>2</sub>CO<sub>3</sub>, 0.2% KOH, NH<sub>4</sub>OH, alc. KOH, acid alc., acid ether and (NH<sub>4</sub>)<sub>2</sub>S while melanin is not changed by any of these. Spectroscopic exam. of a soln. of malarial pigment proved conclusively that the pigment is hematin. It is suggested that the action of a proteolytic enzyme of the malarial parasite upon the hemoglobin of the red cells is the most probable source of malarial pigment. The difficulty with which the human organism disposes of malarial pigment indicates that the production of hematin cannot be considered as a normal intermediate process in the formation of bile pigments from hemoglobin.

Walter A. Jacobs.

The Value of Hydrogen Peroxide in the Microchemical Determination of Iron. W. H. Brown. Path. Lab., Univ. of Wisc. J. Exp. Med., 13, 477-85.—In soln. or within the red cells, hemoglobin can be oxidized by  $H_2O_2$  to form a colorless substance giving the microchem. Fe reactions, the type of reaction depending upon the degree of oxidation. Moreover, hematin and hemin are decolorized by  $H_2O_2$ . The colorless substance preserved the form of the original and also gave the Fe reactions. A method is described for obtaining the microchem. Fe reaction from hemoglobin and its various modifications and Fe-containing derivs., from tissue cells, from cell nuclei, and from eosinophil granules, by an appropriate degree of oxidation with  $H_2O_2$ . W. A. J.

The Quantitative Estimation of the Indophenol Oxidase of Animal Tissues. H. M. VERNON. Physiol. Lab., Oxford. J. Physiol., 42, 402-27.—The formation of indophenol by the oxidation of a mixt. of  $\alpha$ -naphthol and  $\beta$ -phenyl-diamine under the influence of minced tissues, has been detd. quant. The amt. produced does not follow under all conditions any one law but varies with several factors. (i) It may be in direct linear proportion to the time, but as a rule the reaction gradually slows down. (2) With a substrate containing 0.01 M conc. of  $\alpha$ -naphthol and diamine it is proportional to the square of the quantity of the enzyme (law of Medwedew). (3) With 0.006 M substrate it is directly proportional to the quantity of enzyme (law of Schütz). The oxidasic power of the tissues runs parallel to their degree of O satn., and it is provisionally suggested that this O is stored up in the tissues intramolecularly in the form of organic peroxides or oxygenase. The activity of the oxidase is affected by the temp. like other enzymes, but it is very unstable at temps, above 30°.

J. F. LYMAN.

A Method for Estimating Acetone in Animal Liquids. H. Scott-Wilson. Physiol. Lab., Oxford. J. Physiol., 42, 444-70.—The pptn. of acetone by  $Hg(CN)_2$  in KOH as described by Marsh and Struthers (J. Chem. Soc., 87, 1878) has been made quant. by substituting an alk. soln. of basic  $Hg(CN)_2$ . The reaction is:  $C_2H_4O + 2 Hg(CN)_2 + 3 HgO = C_2OHg_4C_4N_4 + 3 H_2O$ . The compd. formed is insol., it can be sepd. by filtration and the Hg content detd. by titration with stand. KSCN (1 mg. Hg = 0.058 mg. acetone). 100 cc. urin (or less if the acetone content is abnormally high) are put in a flask with 25 g. anhydrous  $Na_2SO_4$  and 1 cc.  $H_2SO_4$ . The mixt. is then distilled, the steam being passed through a b. 40% soda soln. and then condensed, the end of the condenser dipping below the surface of the pptg. reagent in the receiving flask. The pptd. Hg compd. is sepd., the acetone oxidized by KMnO<sub>4</sub> in acid soln., and the Hg detd. The reagent contains:  $Hg(CN)_2$  0.5 g., NaOH 9.0 g.,  $H_2O$  60 cc.,  $AgNO_3$  (0.7268% soln.) 20 cc. The detn. may be carried out on as little as 20 cc. normal

urin. The time required is 40 min.; the error is less than 0.006 mg. of acetone. Diacetic acid is detd as acetone by the method. The av. daily excretion of free and combined acetone in human urin is 2-4 mg. A parallelism is pointed out between the N and acetone excreted per diem. A method is given for detecting the presence of abnormal amts. of acetone in expired air.

J. F. LYMAN.

The Determination of the Constants of the Differential Blood-gas Apparatus.

JOSEPH BARCROFT AND H. L. HIGGINS. Physiol. Lab., Cambridge. J. Physiol., 42, 512-8.—A graphic method and a method of simultaneous equations are both applied for the detn. of the constants of blood-gas apparatus.

J. F. L.

Clinical Estimation of Amino Acids. CARLO ROLLA. Med. Klinik, Genua. Pathologica, 2, 575-7; through Zentr. Biochem. Biophys., 11, 893.—When amino acids were given to healthy subjects, to subjects suffering from cirrhosis of the liver, and to subjects suffering from other diseases followed 2 or 3 days later by Sörensen's test for amino acids in the urin and feces, it was found, in general, that in subjects suffering from cirrhosis in proportion to the total N excretion in the urin the amino acid N is present in higher % than in healthy subjects or subjects suffering from other maladies. If the amino acids are administered per os, the excretion can not be increased. Amino acids administered per os can not be found in the feces, hence, they are either changed in the intestin or are there absorbed.

Study and Identification of Pepsin of the Stomach. Calisto Abbo. Med. Klin., Genua. La clin. med. ital., 49, 170-86; through Zentr. Biochem. Biophys., 11, 901-2.

—Criticism of methods followed by results by Barlocco's method. This method depends upon the power of pepsin to clear up 0.05 cc. of a 2% casein soln. acidified with a few drops of a mixt. of 5 g. HOAc, 45 g. alc. and 50 g. H<sub>2</sub>O. The pepsin was diluted with 0.01% HCl and 1 cc. used for every test. The method is considered most simple as well as most delicate and reliable.

I. K. P.

A New Color Reagent for Callose. M. S. TZVYHTT. Compt. rend., 153, 503-5.

—By allowing an alk. soln. of resorcinol to stand exposed to the air, a blue soln. is obtained which when diluted stains callose selectively. It has the advantage over other callose reagents that it can be employed with one or two other stains for other parts of the tissue, the callose blue still showing up distinctly.

H. A. Sporhr.

Determination of Lecithin (VIRCHOW). 12.

Detection of Blood (FURTH). 7.

#### BACTERIOLOGY.

The Behavior of Yeast Enzymes, Free and Combined with Protoplasm. HANS EULER AND SIXTEN KULLBERG. Biochem. Lab., Hochsch., Stockholm. Z. physiol. Chem., 73, 85-100.—I. Zymase. The fermentation of sugar by living yeast is rapidly and almost completely inhibited by CHCl2, toluene, and thymol. Vacuum-dried yeast retains at most 1/10 to 1/30 of its zymase in active condition; its fermenting power, even when it is completely steril, is notably depressed by toluene and thymol. Yeast does not entirely cease to ferment after 1/2 hr. at -180°. II. Maltase. Toluene and thymol diminish greatly the maltase action of fresh yeast; CHCl, inhibits it entirely. The maltase of dried yeast is less susceptible. III. Invertase. After drying, ordinary yeast retains about 50% of its invertase in active form. Antiseptics do not affect at all the inverting power of either the living or the dried yeast. Monilia yeast shows but slight invertase action, while the enzyme is difficult to ext., and is readily poisoned. To explain the above differences the following hypothesis is offered. The yeast enzymes are originally constituents of the cell plasma. Either they are set free within the living cell, to be generated anew by the plasma, in which case they are relatively easy to ext., and are present in the cell in considerable amt. (invertase);

or they are liberated only on drying the yeast, or through other mechanical agencies attended by the death of the protoplasm. They are resistant to the action of antiseptics just in proportion as they are freed from the living plasma. A. HUNTER.

The Significance of Dioxyacetone as an Intermediate Product of Alcoholic Fermentation. S. Karaushanov. Bot. Lab., Univ. Odessa. Ber. bot. ges., 29, 322-7.—
B. Jensen had found that CO<sub>2</sub> and EtOH were formed by the action of animal charcoal on the dihydroxyacetone contained in glycerose. K. shows that Jensen's methods of analysis are not reliable and repeats some of the expts. The glycerose soln. is shown to be acid, and liberates the same amt. of CO<sub>2</sub> from animal charcoal as H<sub>2</sub>SO<sub>4</sub> of equal strength. Repeated efforts to obtain EtOH from a mixt. of animal charcoal and glycerose failed.

The Formation of Nitric Oxide in the Denitrification Process Induced by B. hartlebi. A. J. Lebedev. Agr. Chem. Lab., Univ. Odessa. Ber bot. Ges., 29, 327-9.—NO accompanies the denitrification process of B. hartlebi if (1) the action is allowed to proceed under absolutely anaerobic conditions and (2) with the following nutrient soln.: K Na tartrate 1.0%, KNO<sub>3</sub> 0.3%, KH<sub>2</sub>PO<sub>4</sub> 0.05%, MgSO<sub>4</sub> 0.02%, FeCl<sub>2</sub> trace, with the soln. made slightly alk. with KOH. The presence of NO was established by the immediate oxidation to N<sub>2</sub>O<sub>4</sub> in the air and the oxidation to HNO<sub>2</sub> and HNO<sub>3</sub> in the presence of H<sub>2</sub>O.

H. A. Spoehr.

Is Zymase a Diastase? A. J. LEBEDEV. Bull. soc. chim., 9, 672-82.—Using the juices prepared by the author's maceration method, it is concluded that zymase is a typical enzyme. The amt. of sugar fermented is about proportional to the amt. of coënzyme present, when this is present in a conc. of at least 20% of the active juice. The extraordinary activity of the juice prepared according to the author's method is due to the presence of a large amt. of coënzyme. Yeast has a greater activity than any prepared juice not because the former contains any greater amt. of zymase, but because the yeast cell has the power of synthesizing the coënzyme which is continually used up in the process of fermentation.

H. A. S.

#### BOTANY.

Origin of Osmotic Effects. IV. Differential Septa in Plants with Reference to the Translocation of Nutritive Materials. Henry E. Armstrong and E. Frankland Armstrong. Proc. Roy. Soc. London, (B), 84, 226-9.—When leaves of Aucuba japonica are placed in water containing a substance which can penetrate into the leaf [the particular subtance used is not stated. Abstr.], the leaf changes in appearance and reducing sugar equal to 3-4% of leaf wt. diffuses in 3-4 days. If, however, 0.2% HCN be added, some changes take place but no sugar diffuses. This is supposed to be due to the inhibition of oxidation by HCN. Hence the activity of oxidases alters the septa and causes a translocation of nutriment. Similar effects were noted in other leaves, in roots (radish and beet), in unripe fruits and seed pods.

I. K. Phelps.

Hemaglutins in Plants. M. v. EISLER AND L. v. PORTHEIM. Ber. bot. Ges., 29, 419-30.—The appearance and disappearance of agglutants in seeds is simultaneous with the formation and subsequent utilization of the reserve material.

H. A. SPOEHR.

The Color Changes Occurring in the Blue Flowers of the Wild Chicory, Cichorium intybus. J. H. Kastle and R. L. Haden. Am. Chem. J., 46, 315-25.—During the day the flowers of the wild chicory fade to pink, then white and finally wither and become brown. It has been found that these changes in color are associated with a decrease in the acidity of the cell contents, and that the final browning of the leaf is due to the action of an oxidase which is contained in the flower and completely oxi-

dizes the pigment. Oxidases derived from other plants as well as chem. oxidizing agents show the same destructive effect.

H. A. S.

Influence of the Reaction Medium on the Activity of Cellase. New Characteristic Distinction from Emulsin. G. Bertrand and Arthur Compton. Compt. rend., 153, 360-3.—A prep. of sweet almonds showed slight alkalinity towards methyl orange and slight acidity toward phenolph. This prep. has the power of hydrolyzing cellose a disaccharide obtained from the partial hydrolysis of cellulose. The slightest addition of acid or alkali inhibits the action entirely. The 2 diastases amygdalinase and amygdalase, confused continually under the name of emulsin, differ from cellase in that these show their max. activity in a soln. which is slightly alkaline to phenolph. H. A. S.

Notes on Plant Chemistry. P. Q. KEEGAN. Chem. News, 104, 109-10.—The analysis of a large number of plants shows that starch is but rarely present in leaves in sufficient quantity for detection with the I reagent. Many forms of chlorophyll granules do not manuf. starch nor is it to be found in most leaves as reserve material. True reserve starch exists in roots and medullary rays. Sol. starch was not found in any leaves. The inference is drawn that sugar is an important product of assimilation, that it is independent of starch, is not a respiratory material and is not convertible into acids. It is concluded that syponarin (obtained from Alliaria officinalis) is a tannoid or glucoside of a flavone deriv. Analyses are given to substantiate the theory of Pichard that the presence of chlorides in the soil and their tendency to be absorbed by the plant is antagonistic to the taking up of nitrates.

Action of Nitrosyl Chloride on *Bupleurum fruticosum* (Francesconi, Sanna). 17. Essence of *Bupleurum fruticosum* (Francesconi, Semagiotto). 17.

#### PHYSIOLOGY.

The Action of the Enzymes of the Stomach of the Pancreas and of the Intestinal Mucosa on Gelatin. D. MINAMI. Chem. Abtiel. physiol. Inst., Breslau. Biochem. Z., 34, 248-60.—Pepsin-HCl and the ext. of the mucosa of the small intestin exert only a slight action comparatively on gelatin. Pancreas ext. works more powerfully, forming peptide which could not be pptd. by tannic acid. Small quantities of *l*-leucine and *l*-prolin were found; glycocoll was absent. This latter is of interest since Mesernitzky found considerable quantities of glycocoll, using prodigious gelatinase.

F. J. BIRCHARD.

The Functions of the Liver. E. Wehrle. Allgem. Poliklin., Basel. Biochem. Z., 34, 233-41.—Expts. were made on geese. Although the liver plays an important part in the case of carbohydrates yet after excision of the liver, large quantities of carbohydrates are still assimilated by the organism. Expts. with levulose, dextrose, maltose, saccharose and starch showed that the organism exercizes various degrees of tolerance, particularly in the case of levulose. These relations underwent no essential change after removal of the liver. The diminished tolerance observed in persons suffering from certain diseases of the liver is probably due to disturbances of a general nature—intoxication, decreased powers of oxidation of the organs, fever, etc. Excision of the liver is accompanied by a marked increase in the amt. of NH<sub>4</sub>OH eliminated. The increase of amino N eliminated after feeding glycocoll is greater after removal of the liver.

Anti-coagulants on Frog's Blood. II. H. PRINGLE AND J. TAIT. Proc. Physiol. Soc., 1911, xxxviii-ixl; J. Physiol., 42.—Most of the ordinary anti-coagulants prevent cytolysis of the thrombocytes in frog's blood. The presence of Ca is necessary for the disorganization of these thrombocytes.

J. F. Lyman.

The Presence of Secretin during Fetal Life. H. PRINGLE. Proc. Physiol. Soc., 1911, xl; J. Physiol., 42.—Secretin is present in newly born kittens. An active secretin could not be prepd. from fetuses in all stages of development. J. F. LYMAN.

The Identity of Pepsin and Rennin. AGNES E. PORTER. Lister Inst., London. J. Physiol., 42, 389-401.—Pepsin and rennin are held to be two distinct enzymes because of the following observations: (1) Several com. preps. of rennin were actively milk-coagulating but antipeptic. (2) This antipeptic activity was due to substances indifferent to rennin. (3) These substances could be dialyzed away easily, leaving little or no increase in peptic activity. (4) A rennin powder and several dialyzed rennin preps. were non-peptic, non-antipeptic, while being strongly milk coagulating. (5) A zymoid form of rennin, which was indifferent to pepsin, occurred spontaneously in dialyzed rennin.

J. F. L.

Magnitude of the Work of the Liver. F. Verzar. Physiol. chem. lab., Univ. Budapest. Biochem. Z., 34, 53-62.—About 12% of the total energy metabolism falls to the liver.

G. M. M.

Is Liver Function Essential for the Oxidation of Carbohydrates? F. VERZAR. Physiol. chem. lab., Univ. Budapest. Biochem. Z., 34, 62-5.—Carbohydrates are oxidized even when the liver is excluded from the portal circulation. Oxidation may take place without a previous conversion of the carbohydrates to glycogen.

G. M. M.

Rature of the Sugar in the Blood. F. SPALITTA. Physiol. Inst., Palermo. Arch. ital. biol., 53, 356-62; through Zentr. Biochem. Biophys., 11, 889.—The reducing power of the blood of sea turtles (Thalassochelis caretta) increases when heated with mineral acid. The increase varies in individual animals, being highest in those well nourished and in those freshly taken from sea water. The presence of maltose or isomaltose in the blood is suggested as the explanation.

I. K. Phelps.

Hypotensive Action of the Pancreas. ALBERTO FARINI. Ist. patol. spec. med. Padova. Lo sper., 64, 49-69; through Zentr. Biochem Biophys., 11, 878-9.—Pancreas exts. were injected intravenously into the neck of dogs and cats. This caused in 8-10 sec. lowering of the blood pressure which remained some mins. at the lowest pressure reached before it began to rize slowly to normal. The hypotensive action and the activity of the pancreas exts. varied somewhat. Greater hypotensive power was found in the pancreas ext. than in ext. of thyroid, thymus or spleen. The lowering of arterial pressure seems due to the action of the pancreas ext. causing an antagonistic action of the vasomotor centers and the muscular action of the blood vessels. This hypothesis accounts for the short duration of the hypotensive action of the pancreas ext.

I. K. P.

Inner Respiration of Tissues and its Relation to Death from Hunger and to Acidosis. France Rossi. Physiol. Inst., Bologna. Bull. sci. med., 81, 149-54; through Zentr. Biochem. Biophys., 11, 874.—The results of Lussana are confirmed in showing that CO<sub>2</sub> accumulates in a surviving liver as it does also in inanition to the point of producing an acidosis. Some change in the inner respiration of the tissue has taken place, the CO<sub>2</sub> not being taken away by the blood acts on the respiratory center and on the nervous tissue to produce stimulation at first, and afterward depression. I. K. P.

Action of the Products Formed by Work in Muscle on the Nerve Centers. ALBERTO GRAZIANI. Hyg. Inst. Padua. Ann. ig. sper., 20, 343-70; through Zentr. Biochem. Biophys., 11, 871.—Muscle ext. from a fatigued animal was mixed in known amt. with cerebral substance. The toxicity of this ext. was found to be greater than the ext. from the muscle of a resting animal. The substance of the nerve center has the power of binding the toxic substances in the muscle ext. While the muscle

also has the power of binding the toxic substances, but to a lesser extent, the nerve substance holds the toxic substances and suffers directly.

I. K. P.

Electrical Conductivity of Mixed Human Saliva. GIOVANNI POLARA. Physiol. Inst. Catania. Arch. farmacol., 9, 350-7; through Zontr. Biochem. Biophys., 11, 882-3.—The elec. cond. and the osmotic pressure of his own saliva were detd. at different hrs. of the day to find to what extent the mol. conc. of the saliva is influenced by food. P. found the largest amt. of ionizable salts in the morning, decreasing gradually to be uninfluenced by light meals. The total mol. conc. was greatest in the morning, decreasing until the evening dinner, when it again increased. Substances which ionize occurred in the saliva only after hearty meals. The saliva always showed a neutral reaction.

I. K. Phelles.

Nascent Oxygen and its Anticoagulating Power on Fibrinogen and Fibrinolytic Substances. Corrado Bernabel. Inst. spez. med. Pathol. Sienna. Arch. fisiol., 8, 458-62; through Zentr. Biochem. Biophys., 11, 889-90.—The action of O on the blood plasma is to check the coagulation of the fibrinogen. If H<sub>2</sub>O<sub>2</sub> is added to fibrin, the fibrin is broken up, becoming a fluid, colorless mass of increasing acidity in which serum-albumin, serum-globulin, proteoses and peptones are produced. If fresh fibrin is first heated to 90°, then treated with H<sub>2</sub>O<sub>2</sub>, the catalase and the fibrinolytic action is lessened. By adding H<sub>2</sub>O<sub>3</sub> directly to blood in toto it was not possible to follow the anticoagulating and fibrinolytic action, yet by the addition of catalase and red pigment of the serum a soln. of the clot could be caused. It is concluded that the O activated by the oxidase performs an important anticoagulating function in the blood.

I. K. Pheles.

Group of Substances Indicated by some Tests on Urin. O. CANTELLI. Bull. sci. med., 81, 166-75; through Zentr. Biochem. Biophys., 11, 887.—The presence of 4 substances not previously known in urin was detected. Of these, 3 appear as a ppt. on heating gently the urin alone or with dil. HOAc. Alc. or Et<sub>2</sub>O cannot prevent this pptn. Urins containing these substances on diln. and addition of glacial HOAc give the nucleoprotein reaction. The 4th substance is pptd. in the cold with glacial HOAc, differing from the mucins by appearing after a time. One of the 3 substances gives with Zn dust and HCl a red color after isolation from the urin and resoln. Another is pptd. by heating in presence of glacial HOAc. A third is pptd. from the soln. in glacial HOAc by chilling. All 4 substances are pptd. by picric acid, trichloroacetic acid and Esbach's reagent. They do not react with fuming H<sub>2</sub>SO<sub>4</sub> or citric acid. They give the biuret test after extraction.

HAMMARSTEN, O.: A Text-Book of Physiological Chemistry. Translated from 7th Ed. by John A. Mandel. New York: John Wiley & Sons. 8°, 964 pp., \$4.00.

#### PATHOLOGY.

Physical and Chemical Changes Taking Place in the Blood during Starvation.

MICHAEL POLANYI. Physiol. chem. lab., Univ. Budapest. Biochem. Z., 34, 192204.—The protein content of serum is lowered by starvation. This causes a diminution of the solids, n, viscosity and d. of the blood. The surface tension is increased. The ash content (also Cl) of the serum increases. No definit change could be established with reference to the fat content. H+ conc. increases.

H. J. Corper.

Experiments on Fat Formation in Implanted Organs. WM. GRIESSER. Path. anat. Inst., Univ. of Freiburg. Beitr. path. Anat., 51, 115-36.—G. concluded from expts. with implanted organs that the implanted organ is not the source of the fat deposition which occurs in the zone of organization, but that it is more likely a fat transportation occurring from outside. The intensity of fat deposition conformed well with the leucocytic infiltration that surrounded the implanted organ. The expts.

consisted of implanting into rabbits, fatty organs (kidney and liver) obtained from white mice which had been given P in oil emulsion by injection; physiological fatty organs (kidney and testes) of old cats implanted under the skin and in the abdominal cavity of the same animal; organs (kidney and liver) from phosphorized mice, thoroughly extracted with alc. and ether, placed in the abdomen of rabbits, and like organs fixed by "formol" instead of treating with alc. and ether; and physiological fatty organs (kidney and testes) of cats fixed with "formol."

H. J. C.

Ochronosis in Animals. ALEXANDRA INGIER. Path. Inst., Friedrichstädter Kranken-haus., Dresden. Beitr. path. Anat., 51, 199–208.—I. examined 2 cases of brown pigmentation of the skeletal system, one a 2 yr. old ox and the other a 1 yr. old pig. Microscopically the pigment was granular in form (nowhere cryst.) and with no predilection as to localization in the ground substance. The bone tissue itself showed no abnormalities. In the bone marrow, especially the spongy part, large amts. of pigment were seen, partly free but the greater part in large lymphocytes (myeloblasts) accumulated especially around the nucleus. The pigment gave no microchem. Fe reaction in this case and spectroscopical exam. of acid and alkali exts. of the bones revealed the absorption bands of such solns. of hematoporphorin. Water, alc., ether and chloroform solns. remained uncolored. The disease is a metabolism anomaly, the pigment belongs to the melanin group, and is possibly a deriv. of chlorophyll.

H. J. C.

The Composition of Human Milk in Nephritis. ST. ENGEL, AND H. MURSCH-HAUSER. Akad. Kinderklinik, Düsseldorf. Z. physiol. Chem., 73, 101-8.—The milk and blood serum of a 21 yr. old primipara with chronic nephritis (urinary albumin — 0.3%) were simultaneously examd., upon 2 occasions. The "rest-N" of the serum was 5.33 and 7.43% of the total N; that of the milk 27.19 and 32.88%; both much above normal. The responsible substance is probably urea. The milk secretion as a whole and in its general comp. was unaffected.

A. HUNTER.

Chemical Study of the Brain in Cases of Dementia Practox. W. Koch. Path. Lab., Claybury and Long Grove Asylum, Epsom. J. Exp. Med., 13, 301-7.—Chemical analyses of S in the different fractions of the brain in cases of dementia practox showed variations from the normal in protein S of 1-10%, in lipoid S of 1-17%, in neutral S of 27-65% and but little variation in inorganic S.

Walter A. Jacobs.

Plaques and Coagulation. G. VINCI AND A. CHISTONI. Inst. exper. Pharmacol., Neapel. Arch. ital. biol., 53, 206-12; through Zentr. Biochem. Biophys., 11, 890.— The blood of Gallus domesticus, Ligurnis chloris, and Columba livia possesses no blood plaques, but coagulates quickly and completely. Dog's lymph, and to some extent that of the rabbit and the cat, has blood plaques, normally, but in experimental lymphorrhea they do not have plaques. Nevertheless the blood coagulates. Although plaques are not necessary for coagulation their presence quickens and intensifies coagulation.

J. K. Phelps.

Diabetes Mellitus. NICOLA DE DOMINICIS. R. Univ., Napoli. Gas. int. med., 1910, 435-7; through Zentr. Biochem. Biophys., 11, 877.—When the ext. of the contents of 12 finger lengths of intestin, taken from dogs with extirpated pancreas, was injected into a healthy dog, glycosuria followed, of similar intensity, and duration as follows an injection of phlorhizin. The transfusion of 200 cc. of blood from the carotid of a healthy dog into the jugular of a dog suffering from glycosuria, in consequence of pancreas extirpation, produced an increase in glycosuria instead of the expected decrease. When large amts. of glucose were injected into the jugular of dogs suffering from diabetes incipidus, following pancreas extirpation, and from inanition to the point of weakness, no trace of the injected glucose was found in the urin. From these facts

and the fact that glycosuria is not produced by ligation of Wirsung's duct, the part which a distubance of the digestion plays in diabetes and in glycosuria is obvious.

I. K. P.

Amylolytic Power of Mixed Saliva in Different Pathological Conditions and in Gastric Disorders in Particular. Gino Pagliai. Riv. crit. clin. med., 11, 341-7; through Zentr. Biochem. Biophys., 11, 898.—The amylolytic power of the saliva varies in different individuals and from time to time in each individual. It increases usually during meals and 2 hrs. later has decreased markedly. In illness its amylolytic power varies with the malady qualitatively and quantitatively. For instance, in such acute illnesses as cachexia it is lessened, while in diseases affecting the stomach it is approx. unchanged. The cause is due rather to a bad condition of metabolism or poor nourishment than to the sp. disease.

I. K. P.

Cathemoglobin and its Appearance in Body Fluids Independent of the Action of Poisons. CEVIDALLI. Inst. Legal Med., Florence. Lo sperim., 64, 438; through Z. Immunität., Ref., 4, 390.—Observed the presence of cathemoglobin in a pseudo-mucinous cyst of the ovary, it having been previously found by Takayama in the urin of poisoned rabbits. This overthrows the opinion that this substance results only from action of certain poisons.

H. G. Wells.

The Excretion of Loosely Bound Iron in Malaria. G. GIRMSA. Inst. Schiffs. u. Tropenkrankh., Hamburg. Arch. Schiffs. u. Tropenkyg., 15, 305.—In all cases of well-defined malaria with demonstration of parasites in the blood, this Fe compound was found in the urin. Its absence from the urin is of no diagnostic value in latent cases.

H. G. W.

Proteolytic Enzymes in Diphtheria and Scarlatina Exudates. G. Guid. Kinder-klinik, Florence. Pathologica, 2, 418-21; through Z. Immunität., Ref., 4, 427.—In diphtheria exudates the proteolytic enzymes are abundant, in scarlatina only slight, and rarely or not at all in simple angina.

H. G. W.

Influence of Phenol upon the Virus of Rabies. V. SAVTSHENEO. L'inst. bacterolog. de Kharkoff. Ann. inst. Pasteur, 25, 492-6.—Phenol in strengths of 1, 2, 3, 4 and 5% did not destroy the virus of rabies in 24 hrs. (tested in rabbits). Phenol of 0.5% strength does not destroy the virus in 28 days and 3 and 5% solns. do not destroy it within 7 days.

H. J. CORPER.

The Composition of Urinary Albumin. FLORENTIN MEDIGRECEANU. New York. J. Exp. Med., 14, 298-305.—An application of the method of Van Slyke (C. A., 5, 509, 2856) to the analysis of urinary albumin from dogs with experimental nephritis and from cases of nephritis in man.

W. A. J.

Viscometry of the Urin. L. BORHLI AND DATTA. Allg. med. Klin., Turin. Riv. crit. clin. med., 11, 289-95; through Zentr. Biochem. Biophys., 11, 886.—From the exam. of pathological and normal urins it was found that viscosity and sp. gr. are independent of each other. Of greater influence is the mucin and the incomplete cleavage products of protein. In acute nephritis viscosity is above normal, in chronic nephritis either normal or slightly below.

I. K. Phelps.

Identification of Pancreas Enzymes in Stomach Contents. UMBERTO CARPI. Med. Klin., Pavia. Gazz. med. ital., 61, 73-4; through Zentr. Biochem. Biophys., 11, 899-900.—The tryptic power of stomach contents was tested after feeding an Ewald test meal. The highest tryptic power was found in cases of achylia, hypo-acidity and cancer of the stomach. Lower values were obtained in hyper-acidity and stenosis of the pylorus. The method for detn. of pancreatic activity was recommended for the simplicity of the technic.

I. K. P.

Intermediate Products of Carbohydrate Decomposition as Irritants to the Liver

in Glucose Excretion. EUGENIO CENTANNI. R. accad. fisiocrit. Siena, 1910, March 20; through Zentr. Biochem. Biophys., 11, 877.—By means of artificial circulation in the living organism and of hashed liver in  $H_2O$  the intermediate products of carbohydrate catabolism were studied, vis., HOAc, acetoacetic acid, lactic acid,  $\beta$ -hydroxy-butyric acid, acetone and alc. A condition of hyperglucemia and glucosuria was produced. This is explained as due to an irritating action of the substances used on the liver cells. The continued action of these irritants can produce any of the forms of diabetes.

I. K. P.

Cultivation of the Leprosy Bacillus from the Human Tissue with Special Reference to the Amino Acids as Culture Media. Charles W. Duval. Lab. of Path., Tulane Univ., New Orleans. J. Exp. Med., 13, 365-73.—In the cultivation of Bacillus leprae the initial multiplication outside the body cannot be obtained unless amino acids are present in the medium. This condition is obtained by the use of tryptophan or a mixt. of albumin and trypsin or indirectly by a bacterial species capable of digesting the albumin constituent of the medium employed.

Walter A. Jacobs.

Chemistry of Hydrocephalic Liquid. MICHAEL POLANYI. Physiol. chem. lab., Univ. Budapest. Biochem. Z., 34, 205-10.—From analytical data, P. concludes that the cerebrospinal liquid is not a transudate but lymph.

G. M. MEYER.

#### PHARMACOLOGY.

Action of Iron on the Mobil Oxygen of the Blood. GIUSEPPE ROCCHI. Univ. Bologna. Arch. farm. sper., 12, 317-24.

A. W. Dox.

The Action of Drugs under Pathological Conditions. W. SALANT, U. S. Dept. Agr., Bur. Chem., Circ. 81.—Expts. indicate that the reaction of a cell to foreign substances may be different under changed conditions of environment or after the production of morphological chemical or physical changes, for a disturbance of any of these factors often brings about a variation in pharmacologic action, and the selective action of drugs is at least quantitatively modified by morbid change. This difference in action of drugs in health and disease may be due to toxins, to inanition, or to a difference in temp. Starved animals succumb more quickly to strychnine, phenol and cocaine, while again P poisoning is delayed in complete inanition and partial starvation may increase the resistance of some animals to poison. Chronic alcoholism retards the demethylation of caffeine and theobromine, and in Pb poisoning, atropine is more toxic than under normal conditions. Fatigue often increases the susceptibility to toxic action. Abnormalities of the organs of circulation modify pharmacologic action; a dose of caffeine large enough to produce a mild stimulating effect in the healthy individual will increase many times the pulse rate and amplitude in collapse. Absorption and elimination should be given careful consideration. As all the processes called into existence by experimental procedure or in the course of disease are far from being understood, predictions as to the action of a drug can at best be only approximate.

H. C. FULLER.

Effect of Salvarsan upon the Heart in Dogs. JOHN AUER. New York. J. Exp. Med., 14, 248-55.—The intravenous infusion of 0.5% acid salvarsan causes a weakening of the heart which is usually fatal. The intravenous infusion of 0.5% alkaline salvarsan causes no marked apparent effect upon the heart, but its factors of safety may be so reduced in some instances that a slight extra strain produces fibrillation and death.

Walter A. Jacobs.

Pharmacological Action of Several Halogen-substituted Imidazoles. KARL GUNDER-MANN. Pharm. Inst., Univ. Wurzburg. Arch. exp. Path. Pharm., 65, 259-83.—The iodoimidazoles and the Br deriv., contrary to the unsubstituted free bases, produce even in small doses, subcutaneously, intravenously or per os marked increase in pulse and respiration. Large doses result in death (paralysis of the respiratory centers).

 $N-\alpha,\beta,\mu$ -tetraiodoimidazole, due to its insolubility in H<sub>2</sub>O and indifferent behavior towards alkali and acid is but little absorbed, as is seen in the I excretion after per as administrations. Tetraiodohistidine anhydride showed no perceptible action. The order of toxicity of some of the imidazoles is  $\alpha,\beta,\mu$ -tribromoimidazole (0.2 g. per as in a 6.5 kg. dog in 2 hrs.),  $\beta$ -iodo- $\alpha$ -methylimidoazole (0.3 g. per as in 10 hrs.),  $\alpha,\beta$ -diiodo- $\mu$ -methylimidazole (0.4 g. per as) and  $\alpha,\beta,\mu$ -triiodoimidazole. Expts. with  $\alpha,\beta,\mu$ -triiodoimidazole showed the retention of 62% I in the rabbit and 73% in the dog. The elimination of the remainder continued for 3 days. Of this 50% was removed within the first day. Expts. with  $N-\alpha,\beta,\mu$ -tetraiodoimidazole showed in to resemble iodole in bactericidal properties.

The Influence of Temperature upon the Sensitiveness of Frogs towards Poisons. Y. Sanno. Pharm. Inst., Univ. Tokio. Arch. exp. Path. Pharm., 65, 325-36.—In expts. on frogs it was found that atoxyl and colchicine though strongly active in warm blooded animals were resp. 0.02 and 0.0025 as strong in frogs at room temp. When the frogs were heated to 37° atoxyl was found to be 12 and colchicine 50 times as active as at room temp.

Walter A. Jacobs.

A Few Observations on the Action of Salvarean upon the Irritability of Merve and Muscle. Don R. Joseph. Rockefeller Inst., New York. J. Exp. Med., 13, 634-7.

—Salvarean is a comparatively inactive drug when applied directly to nerve and muscle tissue of the frog.

WALTER A. JACOBS.

The Irritating Powder of the Japanese Wood, Tagayasan. K. IWAKAWA. Pharm. Inst., Univ. Tokio. Arch. exp. Path. Pharm., 65, 315-24.—From the crevices and hollows of this wood, a yellow powder was obtained which gradually turned brown to dark violet on standing in the air. It contained about 73% chrysophanhydroanthrone (Hesse, Ann., 309, 53). By external application on rabbits' eyes the pure substance produces inflammations such as conjunctivitis, keratitis and iritis. The skin is also affected. Internally it attacks the digestive tract and kidneys. In the organism it is oxidized and appears in part in the urin as chrysophanic acid. To prepare it the fine powder obtained from the wood is extracted several times with hot benzene. On cooling chrysophanhydroanthrone crystallizes and is recryst. from hot glac. AcOH.

The Blood-pressor Action of Pituitrin. (Hypophysis Extract.) RUDOLF KLOTZ. Physiol. Inst., Univ. Tübingen. Arch. exp. Path. Pharm., 65, 348-60.—Whereas in normal animals pituitrin very slightly influences the blood pressure, when injected into animals which have already suffered a lowering of the blood pressure, it produces a marked and enduring rise.

WALTER A. JACOBS.

Formation of Precipitates after the Intravenous Injection of Salvarsan. Don R. Joseph. Rockefeller Inst. for Med. Res., New York. J. Exp. Med., 14, 83-98. —Acid solns. of salvarsan, injected intravenously into dogs and rabbits even in the conc. used in man, produce a ppt. in the blood stream. This ppt. which is easily seen and which is abundantly present in the blood from the right ventricle and lungs, can seldom be demonstrated in blood from the left ventricle and arteries. Intravenous injections of salvarsan in doses which surely produce a ppt. in the blood stream, are not, at least in rabbits, necessarily fatal to the animal. Alkaline solns. of salvarsan, even in strong conc. never produce a ppt. when injected intravenously. Formation of Precipitates in the Blood in Vitro by Acid Salvarsan Solutions. Ibid., 179-80.—A 0.1% soln. of acid salvarsan may be added to blood without formation of a ppt. but a very small amt. of a more conc. soln. (0.5-1% or over) gives a definit ppt. A fairly definit relation, therefore, exists between the conc. of the acid salvarsan soln. used and the quantity and consistency of the ppt. formed in vitro. This may account for several fatal results attending the use of conc. salvarsan solns.

W. A. J.

The Distribution of Bromine in the Organism after Administration of Inorganic and Organic Bromine Preparations. ALEX. ELLINGER AND YASHIRO KOTAKE, Univ. Königsberg. Arch. exp. Path. Pharm., 65, 87-119; cf. C. A., 5, 545.—The author sums up as follows: In agreement with Nencki and Schoumouw-Simanowski (Ibid., 34, 313) it was found that following per os administration of NaBr the organs normally richest in Cl became particularly rich in Br. If the last NaBr dosage occurs 24 hrs. before death the greatest % Br content is found in the blood. The ratio of Br to total halogens is, with few exceptions (bone marrow and fat), greatest in the blood and approx. equal in serum and form elements in agreement with Brenniger (Z. exp. Path., 4, 414; 7, 556). Cinammic ester dibromide, based on the same amt. of Br, produces about the same effect, as NaBr. The Br elimination resembles that in the NaBr expt., though a larger fraction of the Br compared to total halogen is removed earlier. A small amt. is excreted in organic combination. A considerable amt., partly excreted through the mucosa, appears in the feces. The distribution in the organs resembles that with NaBr. The blood contains the largest % Br, also largest Br to total halogen ratio. The form elements are richer than the serum. In the brain the Br is almost or completely ionized. The liver can become in restricted amt. a Br depository. With sabromin (Ca dibromobehenate) the Br content of the blood is much smaller and the physiol. Br action is produced only by doses which harm the kidneys. The absorption of the drug is good. Cutaneous tissue and liver become the important Br depots. The ratio of Br (inorganic and organic) to total halogen is the smallest in the blood. It is larger in the corpuscles than in thes erum. In the urin and the brain, Br is ionized. The lipoid solubility of an organic Br prep, permits no conclusions regarding its distribution in the organism. WALTER A. JACOBS.

The Effects of Potassium Salts upon the Circulation and their Action on Plain Muscle. G. C. Mathison. Univ. Coll., London. J. Physiol., 42, 471-94; cf. C. A., 5, 3698.—K salts injected intravenously produce a primary fall in blood pressure, followed, if the dose be not too large, by a secondary rise. This is due to the action of K salts on the heart. Injected intra-arterially K causes an immediate rise in blood pressure in the decerebrate animal, due partly to an excitation of vaso-motor centers and partly to a direct effect upon the vessel walls. K acts as an excitant to somatic and visceral nerve centers and to plain muscle in the cat.

J. F. Lyman.

Chloroform and Reversal of Reflex Effect. C. S. Sherrington and S. C. M. Sowton. J. Physiol., 42, 383-8.—The CHCl<sub>2</sub> reversal can be traced in reflexes expressed by the skeletal muscles. Bayliss (*Ibid.*, 14, 316) has already shown that the reflex effect of stimulating afferent nerves may be changed by a sufficient dose of CHCl<sub>2</sub> from a rise of arterial blood pressure to a fall.

J. F. Lyman.

The Inactivation of Adrenaline in Vitro and in Vivo. W. CRAMER. Proc. Physiol. Soc., 1911, xxxvi; J. Physiol., 42.—A soln. of adrenaline can be completely inactivated by standing for a few min. in contact with a dil. soln. of CH<sub>2</sub>O. Pituitrin is not thus affected, indicating that it is not a primary or secondary amine. J. F. LYMAN.

The Mode of Action of Tetramethylammonium Chloride. C. R. MARSHALL. Proc. Physiol. Soc., 1911, xxxvii-xxxviii; J. Physiol., 42.—The action of tetra-Me-NH<sub>4</sub>Cl in temporarily inhibiting respiration is mainly due to a paralyzing action on the nerve endings of the respiratory muscles.

J. F. Lyman.

The Action of Chloroform upon the Blood Vessels. J. Argyll Campbell. Proc. Physiol. Soc., 1911, xxxiii-xxxiv; J. Physiol., 42.—CHCl<sub>2</sub> added to Ringer's soln. and perfused through the blood vessels of the limbs, lungs, heart, intestins, spleen, liver, and brain causes vaso-constriction. The vessels of the kidneys are dilated on similar perfusion. If CHCl<sub>2</sub> be added to specially oxygenated, defibrinated blood and perfused through the vessels of the lungs and limbs dilation is produced.

J. F. L.

Influence of Intravenous Salt Injections on Respiratory Exchanges. F. Verzar. Physiol. Chem. Lab., Univ. Budapest. Biochem. Z., 54, 41-52.—Intravenous NaCl injections increase O consumption. The CO<sub>2</sub> is not increased in the same ratio. The respiratory quotient is therefore lowered.

G. M. MEYER.

The Work of the Kidneys and the "Specific Dynamic" Action of Foods. F. Tangl. Physiol. Chem. Lab., Univ. Budapest. Biochem. Z., 34, 1-40.—Of the total energy metabolism 7.9% is due to the kidneys. The work of the kidneys (42-45 g.) equals 20 cal. per min. This value is far greater than can be ascribed to the osmotic pressure. Administration of urea or salt increases the energy metabolism. Intravenous injections of NaCl, urea and proteins increases the O consumption even though the kidneys are excluded from the circulation.

G. M. MEYER.

The Sense of Smell. R. FOERSTER. Wockschr. Brau., 28, 356-9.—The nature of the sense of smell and its relation to certain physical characteristics and manifestations is treated. The olfactory nerves are temporarily rendered more sensitive by the action of cocaine in its first stages, and by the administration of strychnine. A bibliography covering works on the sense of smell is appended.

C. A. NOWAK.

Influence of Lactic Acid on the Functions of the Respiratory Center. Ernesto Signorelli. Inst. alig. Path. Neapel. Lo sper., 64, 506-16; through Zentr. Biochem. Biophys., 11, 892.—The mode of introduction of lactic acid into the blood influences very markedly the effect on the respiratory center. The results depend entirely upon the conc. of H ions carried by the blood to the respiratory center. I. K. Phelps.

Relation of Pancreas Extracts to Adrenaline Hypertension. Alberto Farini. La clim. med. ital., 49, 259-69; through Zentr. Biochem. Biophys., 11, 879.—A mixt. of pancreas ext. and adrenaline was injected into the veins of the neck of curarized dogs under artificial respiration in order to learn whether the adrenaline is able to check the hypotension due to the pancreas ext., and, further, whether the adrenaline hypertension can be checked by pancreas ext. It was found that the pancreas ext. was not able to paralyze the active substance of the suprarenals. Adrenaline hypertension was less pronounced when the mixt. was introduced than when adrenaline was introduced alone. A similar action with ext. of the brain, of the thyroid, and of the spleen was found. Adrenaline, however, had a more pronounced hypertensive action, the weaker the hypotensive action of the ext. introduced.

I. K. Phelips.

Phytin Phosphate and Glycerophosphate. II. Effect on Muscular Activity. Ivo Novi. Pharm. Lab., Bologna. Mem. r. accad. sci. Bologna, 6; through Zentr. Biochem. Biophys., 11, 871.—Injection of 1% aq. phytin soln. into the dorsal lymph sac of the frog increases its muscular activity 2-4 times above normal. CaCl<sub>2</sub> present in double the proportion of Ca found in the 1% phytin soln. has no influence. MgCl<sub>2</sub> present in double the proportion found in the phytin soln. increases the activity 2-4 times above normal. A mixt. of CaCl<sub>2</sub> and MgCl<sub>2</sub> in the proportion given above in 0.75 NaCl soln. increases the muscular activity as does MgCl<sub>2</sub>. In 1% soln. of Na glycerophosphate there is no influence at all or only a very slight one but, if Ca glycerophosphate is present under similar conditions, the activity is doubled. Even in that case the activity is not so great as that which phytin produces.

I. K. Phelps.

Liquids which Preserve the Functions in Surviving Tissues. III. Action of some Solutions of Lipoids on the Survival of the Nervous System of Frogs. Gaetano Viale. Physiol. Lab., Turin. Arch. fisiol., 8, 537-70; through Zentr. Biochem. Biophys., 11, 872.—The vitality of a prep. perfused with Ringer's soln. depends upon the temp. and upon the time of year. It increases in the autumn, reaching a max. in Nov., then decreasing until in summer it reaches its lowest value. The curve of vitality corresponds exactly with the content of glycogen. The vitality is increased by adding to the perfusion

liquid, sea water made isotonic, sea water containing urea, Et urethane, acetamide, or chloralhydrate.

I. K. Phelps.

Exchanges in Rats in Parabiosis. G. Amantea and P. Manetta. Physiol. Lab., Rom. Arch. ital. biol., 53, 432-8; through Zentr. Biochem. Biophys., 11, 874.—KI, K<sub>4</sub>FeC<sub>4</sub>N<sub>6</sub>, methylene blue, or phlorhizin were administered per os to 1 of 2 white or spotted rats in parabiosis. The substances administered were found in the urin of both rats, even when the rats had been but recently joined. The appearance in the urin of the substance administered was quicker and more abundant in the animal fed in the case of methylene blue and phlorhizin; with KI the excretion began at about the same time and in about the same amt.

I. K. Phelps.

Thebaine, Morphothebaine, Thebenine and Certain of its Derivatives. Herm. Hildebrant. Pharm. Inst., Halle. Arch. exp. Path. Pharm., 65, 54-8.—Thebaine excites the reflexes of cold-blooded animals but on dogs it exerts a narcotic and antemetic effect similar to that of morphine and chloromorphide. Morphothebaine has an emetic action on dogs but it is weaker than that of apomorphine. Thebenine and methylthebenine reduce the excitability in frogs and lack the antemetic action of thebaine. Thymylmethyldiethylamine, HOC<sub>6</sub>H<sub>2</sub>MePr\*CH<sub>2</sub>NEt, causes convulsions in rabbits whereas dihydroxyphenylethanolamine, (HO)<sub>2</sub>C<sub>6</sub>H<sub>6</sub>CH(OH)CH<sub>2</sub>NH<sub>2</sub>, resembles thebenine in its general action.

J. BISHOP TINGLE.

Action of some Plant Enzymes on Uric Acid. G. Bert. Inst. physiol. Chem., Pavia. Biochem. ter. sper., 2, 266-75; through Zentr. Biochem. Biophys., 11, 902.—The action of papain, of takadiastase, and of malt diastase on uric acid dissolved in Na phosphate, showed that the enzyme had a uricolytic power, although all of the uric acid could not be made to disappear.

I. K. Phelps.

Lactic Acid and Cardiac Muscle. W. BURRIDGE. Proc. Physiol. Soc., 1911, xli; J. Physiol., 42.—The action of lactic acid on the rhythmical properties of the frog heart can be accounted for as being due to the displacement of K and Ca salts. J. F. LYMAN.

The Action of Oxazine (Triaminophenazonium hydrochloride) on Trypanosomes. A. Laveran and Roudsky. Compt. rend., 153, 226-9.—Oxazine has an affinity for centrosomes of the trypanosomes both in vitro and in vivo. The virulence of trypanosomes subjected to the action of oxazine is much diminished. Oxazine injected into normal mice, under 20 g. wt., is lethal in doses of 0.00025. In mice with trypanosomes it is less toxic.

G. M. Meyer.

Venom Poisoning. MAURICE ARTHUS. Compt. rend., 153, 482-4.—The venoms of (1) Naja tripudicus, (2) Crotalus adamanteus and (3) Vipera russellii when injected into rabbits in like doses, 1 mg. in conc. of 1: 1000, give rise to different toxic symptoms. With (1) the effects are similar to curare. With (2) death is caused by a lowering of blood pressure. (3) Death is caused by intravascular coagulation. If (1) is injected in 4 mg. doses and respiration kept up artificially then death is due to arterial depression as with (2). When (2) is injected in 5 mg. doses, death is caused by intravascular clotting. Coagulation may be prevented with (3) if the 1 mg. dose is injected in fractions. Death then results the same as with (2). These expts. demonstrate a property of snake venoms which A. terms pseudo-curarization. Curare action is sp. to (1) and allied species. There are 2 distinct classes of snake venoms, those which curarize and those which do not. Between these are numerous intermediary types. G. M. M.

MEYER, H. H. AND GOTTLIEB, R.: Die experimentelle Pharmakologie als Grundlage der Arzneibehandlung. 2 Aufl. Wien: Urban & Schwarzenberg. 14 M.

ZOOLOGY.

Contribution to the Biochemistry of Protozoa. THEODOR PANZER. Z. physiol. Chem., 73, 109-27.—From 5 swim-bladders of Gadus virens Kohler, were collected

516 g. of a yellow, slimy, cream-like mass, a pure culture of Goussia gads. It contained H<sub>2</sub>O 85.93%, solids 14.07%, N 1.25%, inorganic 1.20%. A dil. NH<sub>2</sub>OH ext. of 100 g. yielded on acidification with AcOH a moderate P-free ppt. of glycoprotein. Of the remainder 370 g. were extd. successively with 95% alc., Et<sub>2</sub>O, cold H<sub>2</sub>O, very dil. NH<sub>4</sub>OH, 0.1% HCl, 0.1% KOH and b. H<sub>2</sub>O. The first 2 exts. yielded traces of a base, needles, and of a phenol-like substance, together with considerable amts. of free fatty acids (mol. wt. 224.8, I no. 41.4), and "neutral fat." The "neutral fat" fractions amounted to 4.9 g., of which the "unsaponifiable residue" (2.02 g.) yielded 1.48 g. cholesterol and 0.54 g. of other alcs. insol. in H<sub>2</sub>O. The cholesterol was partly free, partly in esters. The cold aq. ext. contained traces of glycoprotein, non-coagulable protein, glycogen (?), and mannitol (?). Hot H<sub>2</sub>O dissolved a considerable amt. of a protein with the physical properties of gelatin. The final residue consisted entirely of unaltered spores (3/2 of it capsules). They were dissolved in cold 2% KOH. On addition of AcOH the capsule substance was pptd. It contained no P, S, nucleic acid or chitin, and was apparently an albuminoid of relatively simple constitution: the biuret reaction was positive, Millon and xanthoproteic feeble, glyoxylic and Molisch absent. The filtrate contained 2 proteoses, probably of secondary derivation. The nature of the native proteins of the cell is not clear. Remarkable is the entire absence of nucleoprotein and its derivs. A. HUNTER.

Mucin of the Snail's Foot. C. CAVALCASELLE. Pharm. Lab. Padua. Arch. farmacol., No. 5; through Zentr. Biochem. Biophys., 11, 866-7.—Mucin from the snail's foot, like that of the umbilical cord after treatment with HOAc, is separable into 1 insol. and 2 sol. substances. One of the sol. substances is pptd. by neutralizing the acid; sol. in a faint excess of alkali, showing a similarity to 'omphalin B' of the umbilical cord and the corresponding substance of the synovial mucin. The other sol. substance is pptd. warm by FeCl, and, hence, corresponds to the similar component of the umbilical cord and the synovia. This mucin, however, in insol. substance and total mucin has about the same high % of N, varying from the other mucins. Further, the mucin of the snail's foot fails to have the substance which after neutralizing the acid coagulates with heat.

### 11b. IMMUNOCHEMISTRY.

#### H. GIDEON WELLS.

The Antigenic Function of Tuberculin. A. CALMETTE AND L. MASSOL. Compt. rend., 153, 420-2.—The antibodies found in vaccinated animals (and in considerable amt. in the serum in grave tuberculosis), and whose presence can be detd. by the Bordet-Gengou reaction, do not modify the toxicity of tuberculin for tuberculous animals, the serum having no more protective power in tuberculous guinea pigs than an equal amt. of normal serum. This observation suggested a further study of inhibitrice, a substance found in certain sera of hypervaccinated animals and in tuberculous subjects of good health, which has a greater avidity for Koch's tuberculin and the tubercle bacillus than the antibodies included in the same serum, therefore also masking the Bordet-Gengou reaction. All tuberculins are not equal in antibody binding value; the order of avidity is as follows: (1) Entire bacillus; (2) old tuberculin of Koch; (3) watery ext. of the bacilli. Alc. purified tuberculin and that obtained by evapg, the bacillus-free culture possessed no power of fixing either the inhibitrice or the antibody. Peptone exts. the larger part of the inhibitrice and antibody; the authors, therefore, recommend for titrating the serum of tuberculous subjects, a stand. made by macerating 5 g. of dry bacilli in 100 cc. of 10% Witte's peptone at 60% for 48 hrs. H. J. CORPER.

Comparative Value of the Biological Reactions of Serum Precipitins and those of the So-called Erythroprecipitins in Medico-legal Examination of Blood. D. Mirto. Ist. Med. Leg., Univ. Catania. Arch. farm. sper., 12, 145-58.—For medico-legal purposes, the serum precipitin reactions are more satisfactory than those of erythroprecipitins, since: Serum precipitins are sp. for species only, while erythroprecipitins are sp. for both species and organ; pptg. antisera still give a positive reaction when the absorption bands of oxyhemoglobin can no longer be detected; the prep. of pptg. sera is much more simple than that of erythroprecipitating sera.

A. W. Dox.

Chemo-immunological Studies on Localized Infections. I. Action on the Pneumococcus and its Experimental Infections of Combined Sodium Oleate and Antipneumococcus Serum. RICHARD V. LAMAR. Rockefeller Inst., New York, J. Exp. Med., 13, 1-23.—Very dil. solns. of the alk. oleates without affecting the morphology or reproductive power of the pneumococci, produce such profound changes in their structure as to render them more subject to autolysis and to serum lysis. The serum lysis of the soaped organisms tends to be incomplete with normal serum and to be perfect with an immune anti-pneumococcus serum. The inhibition of their activity which the soaps ordinarily suffer in the presence of protein can be prevented by adding H<sub>2</sub>BO<sub>2</sub>. Infection can not only be prevented when the mixt. of immune serum, soap and boric acid is added to the pneumococcus before injection into the peritoneal cavity of small animals, but likewise when the injection of these 3 substances is made to follow the inoculation of normal highly virulent diplococci. II. Lysis of the Pneumococcus and Hemolysis by Certain Fatty Acids and their Alkali Soaps. Ibid., 380-6.—The higher members of the oleic series are very much more strongly hemolytic than crotonic acid. The action of the latter is probably due solely to its acidity. The soaps of the higher acids, due probably to their greater solubility, are all more active than the free acids. Among the higher acids, there is a close relationship between I no. and hemolytic strength, the higher the one the greater the other. Configuration bears a relationship to hemolytic activity, since chaulmoogric acid, isomeric with linoleic, though taking up only 2 halogen atoms, is less active even than oleic acid. The greatest activity is shown by K linolenate, having a large C chain, straight chain structure, high I no. and good solubility. There is a close parallel relationship between the lytic activity for red blood cells and for virulent pneumococci.

WALTER A. JACOBS.

The Action of Morphine on the Heart. A. A. J EGMOND. Pharm. Inst., Reichs Univ. Utrecht. Arch. exp. Path. Pharm., 65, 197-213.—Very small amts. of morphine (0.04 mg. per kg.) produced in dogs slowing of the pulse. This effect arizes from the direct stimulation of the vagus center and is independent of the respiratory effect of the drug. In the process of immunization against the drug this vagus sensitiveness is not reduced, so that in dogs in which 0.23 g. per kg. no.longer produces narcosis or vomiting, 0.0001 g. per kg. still produces perceptible pulse retardation. This then is a good indicator in "immune" animals of the presence of active amts. of the drug in the organism. The increased tolerance for morphine, then, would not exclusively be due, as Faust has shown, to increased destruction of the drug by the organism. W. A. J.

Comparative Merits of Various Complements and Amboceptors in the Serum Diagnosis of Syphilis. Hideyo Noguchi and J. Bronfenbrenner. Lab. Rockefeller Inst., New York. J. Exp. Med., 13, 78-91.—Expts. on various animals show that for fixation tests guinea pig complement is most favorable. It is the most active and durable of those studied. The rabbit is the best animal for producing antihuman amboceptor. The guinea pig produces a good amboceptor but its small size renders it second in choice.

Walter A. Jacobs.

Variations in the Complement Activity and Fixability of Guinea Pig Serum. HIDEYO

NOGUCHI AND J. BRONFENBRENNER. Lab. Rockefeller Inst., New York. J. Exp. Med., 13, 69-77.—The expts. demonstrated that the complementary activity varies within a definit limit in different specimens of guinea pig serum. The amt. of serum fixed by given const. quantities of syphilitic serum and antigen varies much more markedly than the variations in their complementary activity. These facts demonstrate the irregularity of guinea pig serum in the Wassermann reaction. The authors advise the use of a mixt. of sera from more than 2 animals to reduce this irregularity.

WALTER A. JACOBS.

The Interference of Inactive Serum and Egg-white in the Phenomenon of Complement Fixation. Hiddyo Noguchi and J. Bronfenbrenner. Lab. Rockefeller Inst., New York. J. Exp. Med., 13, 92-7.—The effects of various inactivated or spontaneously deteriorated sera and egg-white were studied. Varying quantities of each serum and egg-white in 20% diln. in 0.9% NaCl soln. were added under 3 different conditions to the fixing mixt. of syphilitic serum and antigen. They were added and incubated (1) before the addition of complement (2) at the same time and (3) after the complement had been fixed. In (1) it was found that the fixing capacity of the serum and antigen mixt. was satd. so that it no longer could fix fresh guinea pig complement. Exceptions were encountered with human and goat sera. In (2) there was somewhat less interference than in (1). In (3) there was no hemolysis. The results of a parallel series of expts. on the complement fixation by sp. ppt. (Bordet-Gengou phenomenon) harmonized perfectly with those of the syphilis reaction.

WALTER A. JACOBS.

Biochemical Studies on So-called Syphilis Antigen. HIDEYO NOGUCHI AND J. Bronfenbrenner. Lab. Rockefeller Inst., New York. J. Exp. Med., 13, 43-68. -The liver tissues of man and certain animals yield upon alc. extraction, various substances, but comparatively few possess antigenic properties. The substances contained in the first alc. ext. were fractionated into 4 groups: (a) substances insol. in ether and hot alc., (b) those insol. in ether but sol. in hot alc., (c) those sol. in ether, alc. and acetone and (d) those insol, in acetone. (d) contains the phosphatides and should be selected for the antigen. In (d), a high antigenic value is associated with a high I value of lipoids obtained from the liver and heart. This rule does not apply to the corresponding fraction from the brain. For the prep. of a standard antigen for Noguchi's method for the diagnosis of syphilis proceed as follows: As a stock soln. an ethereal or alc. 3% soln. of an acetone-insol, fraction is made from the liver or heart. An emulsion is made from 1 vol. of this and 9 vols. 0.9% NaCl soln. If this emulsion is hemolytic or anticomplementary in the dose of 0.4 cc. it is unsuitable, otherwise it is tested for its antigenic strength. If it produces complete inhibition of hemolysis with one unit of syphilitic antibody in doses of 0.02 cc. or less it is suitable. In the fixation test o.1 cc. of the emulsion is used. The stock soln. retains its properties for a long time. WALTER A. JACOBS.

Opsonin Reaction. HANS KRONBERGER. Freiburg i/B. Z. exp. Path., 9, 87-96; through Chem. Zentr., 1911, 11, 53.—K. succeeded as follows in improving the method for the detn. of opsonins: The detn. is performed on the defibrinated blood of the patient removed during digestion-leucocytosis and also on that of a control. The preps. of emulsions of the bacteria in question were made from fresh, virulent pure cultures. For staining the prep. a preserving and thorough fixing process was used. The detn. of opsonic indices proceeded by the counting of series prepared at intervals of 10, 20, 30 and 60 min. from the blood-bacteria mixt. of the patient and control. By this method bacteria (anthrax and tubercle bacilli) showed themselves to be chemotactic towards neutrophile leucocytes with decided alk. chromatin reaction, negatively chemotactic towards lymphocytes with acid reaction of the nucleus.

The transition forms show, corresponding to their chem. character, a diminished power for phagocytosis. Staining differences are given.

WALTER A. JACOBS.

The Value of the Serum of Animals with Experimental Anemia in the Regeneration of Blood. CAMILLO GIBELLI, Inst. path. Chirurg., Univ. Genoa. Arch. exp. Path. Pharm., 65, 284-302.—Serum which is obtained from animals made anemic by bleeding, starvation or by phenylhydrazine, possesses the property of increasing the no, of red cells after injection into healthy animals. But in animals in which anemia has been produced no such effect is observed, and this condition lasts until recovery. From this G. concludes that the serum of anemic animals develops an hematopoetic activity only in combination intra vitam with normal serum. The proof of this was shown in the property of the serum of healthy animals already injected with the serum of anemic animals to increase the red cells after injection into either normal or anemic animals. During an infection the hematopoetic property of an active serum is destroyed. This is seen in the fact that the serum of an infected anemic animal produces no hyperglobulia when injected into normal animals; further the increase in the red cells fails likewise in infected animals after injection with an active serum, which was obtained from an uninfected animal or from a normal animal previously treated with an active serum. In all infectious processes, even when of an attenuated nature, the failure of this action was observed, so that this fact gives it the value of a biological -reaction. WALTER A. JACOBS.

The Proof of Ricin in Feeding Stuffs. W. Mooser. Landw. Vers. Stat., 75, 107-34.—By intravenous injection of different concs. of ricin, free from albumin, M. prepares an antiserum. The feeding stuffs are tested for ricin by the precipitin reaction. His method differs from that of Miessner (C. A., 4, 614) in that the feeding-stuff is extracted with glycerol and the soln. poured into a soln. of ether and alc. when a ppt. is formed. This ppt. is filtered off, dried and taken up in a salt soln. and then tested for the reaction with the antiserum. After examining a large no. of feeding-stuffs M. claims more positive results than can be obtained with Miessner's method. Ricin is not always destroyed by 90° moist and 130° dry heat.

E. C. LATHROP.

Pancreatic Lipase. IV. The Action of the Serum of Mice Inoculated with Malignant Mouse Tumor. J. A. Shaw-Mackenzie. Proc. Physiol. Soc., 1911, 11-16; J. Physiol., 42.—The power of the serum to accelerate lipolytic power of pancreatic lipase is increased in various phases of tumor inoculation and growth in mice. The same power of the serum was noted in cases of human cancer in which recovery was already complete, and also during the course of the disease. The same property is also found in other pathological conditions. This acceleration is suggested as I factor in the natural defensive processes of the body.

J. F. Lyman.

The Source of Immune Substances in Lymph. The Part Played by the Spleen in the Formation of Immune Substances. A. B. Luckhardt and F. C. Becht. Proc. Am. Physiol. Soc., 1910, 11-12, 16-17; Am. J. Physiol., 27.

J. F. L.

The Relation of Death from Burns to Anaphylactic Shock. M. HEYDE. Zentr. physiol., 25, 441-4.—The substance extracted from the urin of animals that died through the effect of burns and which gives rise to symptoms of protracted anaphylactic shock has been identified with Me guanidine.

G. M. MEYER.

Influence of Iodoform, Chloroform and other Fat-soluble Substances on Phagocytosis. H. J. Hamburger, J. de Haan and F. Bubanovic. K. Akad. Wetenschappen, 1911, 894-914 (Dutch); through Chem. Zentr., 1911, I, 1868.—Suspensions of horse leucocytes in 0.9% NaCl were mixed with aq. solns. of CHI<sub>2</sub>, ChCl<sub>2</sub>, C<sub>4</sub>H<sub>2</sub>, camphor, chloralhydrate and turpentine and the phagocytic power of this mixt. detd. according to a previously described method (C. A., 5, 1298). All these substances

accelerate phagocytosis very decidedly provided they are not present in too high a conc. CHI<sub>2</sub> possesses an accelerating influence in dilns. of 1:5,000,000. CHI<sub>2</sub> CHCl<sub>2</sub> and C<sub>6</sub>H<sub>4</sub> also have the power of restoring phagocytic power to leucocytes which have lost this power by being kept at low temp. Camphor does not possess this property; in fact phagocytosis does not take place in the presence of camphor. The results of this investigation show a remarkable analogy with the results of J. Loeb and R. S. Lillie with respect to parthogenesis. The lowering of the surface tension, the amoeboid movements of the plasma meeting with less resistance, is the responsible factor.

G. M. Meyer.

Preliminary Report on the Transmission of Hemolysins from Mother to Offspring. L. W. Famulener. Research Lab., Dept. Health, N. Y. City. Proc. Soc. Exp. Biol. Med., 8, 130-1.—Goats were immunized at different periods during the course of gestation, with washed sheep corpuscles given subcutaneously. At the time of birth, none of the young showed sp. hemolysins in the blood serum. The young getting the colostrum and first milk rapidly acquired, and retained, the sp. antibodies. The colostrum in those cases was very rich in hemolysins, but the antibodies apparently disappeared from the milk output after a few days.

V. C. Myers.

The Relation of the Fixation Reaction to Specific Precipitation. F. P. GAY. Univ. California. Proc. Soc. Exp. Biol. Med., 8, 139-140.—Alexin fixation by a mixt. of serum and antiserum is produced by an antigen-antibody complex distinct from precipitinogen-precipitin, but usually brought down by the ppt. in its formation in such a way as to give the appearance that the fixation is produced by the ppt. itself. Gengou's original supposition, without direct proof, of anti-albuminous sensitizers apparently explains this type of fixation reaction. The best expression of the conditions is, however, found in Nicolle's hypothesis that antisera contain 2 classes of antibodies, "coagulins" and "lysins," the former, in this instance, producing pptn., and the latter, the fixation reaction.

V. C. Myers.

Quantitative Complement Fixation Reaction. B. P. SORMANI. Lab. Onze Liev Vrouwe Gasthuis, Amsterdam. Z. Immunität., 11, 243-63.—Description of method; comparisons, and objects.

H. G. W.

Function of Horse Complement as Antigen. C. Morreschi. Konigl. Inst., Exp. Therapie, Frankfurt a/M. Z. Immunität., 11, 275-87.—Corpuscles bound with amboceptor and treated with horse complement are agglutinated by anti-horse serum, the result of sp. affinity between the complement and certain substances in the anti-serum. Therefore it seems that complement exhibits sp. antigenic character.

H. G. W.

Antibodies to Complement. F. Perussia. Klin. Fortbildungsinst., Milan. Z. Immunitat., 11, 287-94.—By injection of animals with an hemolytic system containing fixed horse complement (corpuscles and amboceptor being from another species) antibodies are obtained reacting with horse complement. Therefore complement is antigenic.

H. G. W.

Brands' Modification of Complement Mid-piece. OLUF THOMSEN AND W. LESCHLY. Stat. Seruminst., Copenhagen. Z. Immunitat., 11, 216-34.—The occurrence of Brand's modification depends upon the degree of diln. of serum by dist. H<sub>2</sub>O. It does not occur when serum is dild. 10-20 times. With diln. of 1-4 the speed of modification depends on the salt conc. and temp. of the globulin soln. Other conditions of modification are also considered, and conclusion reached that it probably is not the mid-piece itself that is modified, but that there is an inhibiting subst. belonging to the euglobulin fraction which is affected.

H. G. W.

Value of the Plastein Phenomenon in Antitrypsin Estimation with Bacterial Proteases.

H. KAMMERER. I. Med. Klin., Munich. Z. Immunität., 11, 235-42.—Bacterial and yeast trypsins show distinctly the phenomenon of plastein formation with casein digestion, and as this occurs at a definit stage of proteolysis it may be used for qualitative demonstration of proteolytic ferments. Antitryptic action of serum inhibits equally plastein formation, and hence plastein formation can be used as index of antitryptic action of serum upon bacterial proteases.

H. G. W.

Anaphylaxis with Streptococci. II. P. TH. MÜLLER. Hyg. Inst., Univ. Graz. Z. Immunität., 11, 200-10.—As in previous expts. the attempt to secure anaphylatoxin from the bodies of streptococci in vitro gave practically negative results. Occasionally active anaphylaxis was secured by the use of large doses. Apparently the slight and tardy production of sp. antibodies is the cause of the exceptional behavior of streptococci.

H. G. W.

Antigenic Properties of Lipoids. III. Experiments with Lipoids of Cestodes. Kurt Meyer. Serobakt. Lab. Stadtkrankenhaus, Stettin. Z. Immunitat., 11, 211-5.—Lipoids of cestodes, insol. in acetone, give reaction of complement-fixation with serum of rabbits immunized with exts. of the substance of the parasites; but these lipoids themselves when injected into rabbits do not cause development of complement-binding antibodies. On the other hand ether extraction of cestode exts. deprives them of antigenic power. It is assumed that the antigen is a lipoid compd., probably a lipoid-protein compd.

H. G. W.

Relation of Complement to Blood Cells Sensitized with Amboceptor. III. Lief-Mann and Cohn. Rudolf. Virchow Krankenhaus, Berlin. Z. Immunität., 11, 166-77. —Hemolytic immune sera vary greatly in their binding of mid-piece. Such binding is not an essential precursor of hemolysis. With moderate sensitization the complement disappears only after it has produced hemolysis, which observation speaks for the fermentative nature of complement. H. G. W.

Critical Observations on the Epiphanin Reaction. O. Kammann. Staat. Hyg. Inst., Hamburg. Z. Immunität., 11, 178-99.—A good review on physical phenomena of immunity reactions. For the execution of the epiphanin reaction of Weichardt ordinary pipets are not suitable; only with the special overflow pipet "Mikra," and with o.1 N or o.01 N solns. can reliable results be obtained. In epiphanin reaction with syphilitic serum (diluted 1: 10) no sp. influence was observed, but the reaction obtained is ascribed to the varying absorptive power of different human serums and their varying content of CO<sub>2</sub> and carbonates. The claim of Weichardt that with more dil. serum sp. epiphanin reactions can be obtained, could not be substantiated.

H. G. W.

Relation of Alexin to Sensitized Bacteria. Ocr. Gengou. Pasteur Inst., Brussels. Z. Immunitât., 11, 143-9.—In contradiction to the conclusions of Skwirsky, G. finds that complement-fixation by sp. ppts. is identical with the fixation by sensitized bacteria or corpuscles. If proper amts. of the reacting substances are used the entire midpiece and part of the end-piece are fixed, while the remainder of the end-piece remains free in the soln.

H. G. W.

Wassermann Reaction in Pathological Anatomy. L. K. Wolff. Path.-Anat. Inst., Univ. Amsterdam. Z. Immunidit., 11, 154-66.—In many cadavers the serum gives positive reactions when there is no syphilis. This is caused by lipoids in the serum, which differ from the substances that cause the W. reaction in syphilis by being removed by BaSO<sub>4</sub>; in this way the source of error is removable, and such treated sera give accurate results as to the existence of syphilis.

H. G. W.

Presence in Blood Serum of Substances which have an Activating or Inhibiting Effect on the Hemolytic Properties of Cobra Venom. C. J. Ross. West Asylums

Research Inst., Glasgow. J. Mental Sci., 57, 34; through Z. Immunitst. (Ref.), 4, 452.—Human sera vary greatly in their ability to activate or inhibit cobra venome hemolysis. Inhibiting sera neutralize the activating sera, but have no influence upon venom-lecithin hemolysis. Ox serum and heated (60°) guinea pig serum inhibit; fresh guinea pig serum activates venom. These effects apparently do not depend upon the lecithin or cholesterol.

H. G. W.

The Biology of the Lens. L. Königstein. Serotherap. Inst., Vienna. Arch. Augenheilk., 68, 414; through Z. Immunitát. (Ref.), 4, 463.—Immunization of rabbits with ox lens produces precipitins, anaphylactins, and complement-binding antibodies. Complement fixation is positive with lenses of ox, horse, rabbit, man, monkey, and chicken, strongest with the first 2. Organ specificity was shown by the absence of fixation with serum from ox, horse, rabbit and chicken.

H. G. W.

Experimental Studies on Toxopeptides. PERCY ZADIK. Konigl. Inst. Infectkr., Berlin. Folia serol., 7, 865-78.—These studies support the work of Wassermann and Keysser, showing that serum may develop toxicity through autodigestion by its own complement, if allowed to stand for 2 days or more; this toxicity decreases on longer standing. Therefore all anaphylatoxin expts. must be controlled to be sure that the poison does not arize in this way independent of the presence of the sp. antigen. The production of anaphylatoxin when kaolin is substituted for the antigen is also confirmed. Immunity to anaphylatoxin, or antianaphylaxis after sublethal doses was not accomplished.

H. G. W.

The Isotoxicity of Normal Guinea Pig Serum. C. VALLARDI. Inst. f. Gewerbe-krankheit, Milan. Folia serol., 7, 879-82.—V. was unable to corroborate the production of toxicity in guinea-pig serum after standing.

H. G. W.

The Relation between Anaphylaxis and Eclampsia. H. Guogishero. Hyg.-Bakt. Inst., Univ. Bern. Z. Immunität., 11, 111-32.—Claims to have positively established the existence in placentas of poisons which produce severe, usually fatal poisoning, when injected intravenously. Other cell-free organ exts. are devoid of this toxicity. G. could find in pregnancy no evidence of anything resembling anaphylaxis against either foetal proteins or the placenta. There is no loss of complement and no precipitin in the maternal blood, and no passive sensitization or anaphylatoxim formation can be obtained. Foetal serum usually contains much less complement than the mother's serum. The poisonous placental extracts reproduce in no way the characteristics of anaphylaxis, not causing dyspnoea or bronchial spasm, not being inhibited by atropin, not causing reduction in complement, not requiring complement to complete the poison, not reducing temp. of poisoned animals and not producing a refractory condition (antianaphylaxis).

H. G. W.

Correction of "Remarks" by A. Biedl and R. Kraus on my Article on "Protein Decomposition Intoxications." H. Pfeiffer. Z. Immunität., 11, 133-42.—Controversial.

H. G. W.

Mechanism of Toxin Action. H. Dold and E. Undermann. Kaiserl. Gesundheitsamt, Berlin. Z. Immunität., x1, 86-102.—True toxins differ from most of the simpler poisons in requiring a much longer period after administration before their poisonous action manifests itself. This is accounted for, in part at least, by the observation that serum must first act upon the toxin for some time before the toxin is activated. Ricin, and the toxins of tetanus and diphtheria, act after a much shorter period of incubation if first digested with fresh guines pig serum 20-24 hrs. or more at 37°. Cobra venom is activated by 2 hrs. digestion with serum. Ricin is also activated by lecithin. Heated serum will not shorten the incubation period. The authors

ascribe the action of fresh serum to solution of the toxins by the lipoids, rendering them more easily able to penetrate the cells and thus more quickly acting.

H. G. W.

Tryptic Digestion of Gram-positive and Gram-negative Bacteria. Paul Wein-goff. Hyg. Inst. Kgl. Tierarztl. Hochschule, Berlin. Z. Immunitat., 11, 1-17.—Com. trypsin (Merck) is more active at 50° than at lower temp., resists alc., CHCl<sub>2</sub> and 1-1000 HgCl<sub>3</sub> soln., and passes through porcelain filters without loss of activity. Bacteria are not digested or reduced in growth or reproduction by this trypsin. When killed with CHCl<sub>3</sub> they also resist trypsin, although undergoing autolysis. Heating bacteria to 60° did not make them digestible, but if heated to 75° the gram-negative forms are almost entirely destroyed by trypsin while the gram-positive forms are still resistant. This difference is not ascribable to endobacterial anti-enzymes, but to the difference in penetrability of the bacterial protoplasm. Leucocytes (guinea pig, aleuronat exudate) also digest bacteria heated to 80°, but not bacteria heated to 60°, which indicates a resemblance between the leucolytic proteoses and trypsin. H. G. Wells.

Thermostability of Mid-piece of Complement. H. K. Marks. Hosp. Rockefeller Inst., New York. Z. Immunität., 11, 18-30.—It was impossible to demonstrate thermostability of the isolated globulin fraction of complement of guinea pigs when dissolved in physiol. NaCl soln. When complement has been heated in its natural environment, however, it may be activated by addition of end-piece, showing that the mid-piece is heat-resistant under these conditions. From such heated serum the globulin fraction may be isolated in an active condition, proving that when in the serum the globulin (mid-piece) portion of complement is thermostable. Sheep mid-piece is thermostable when heated in NaCl soln., although weakened by 55° for 30 min. These observations confirm the theory of complementoid as a resistant haptophore group in serum. Presumably the complementoid is not permanently possessed of an inert toxophore group, or it would not so readily unite with fresh end piece. Some evidence was obtained in favor of the view that the two fractions of complement exist uncombined in the serum. Inactivated end-piece does not interfere with the action of fresh end-piece. Isolated guinea-pig middle-piece is thermolabile when heated in sheep albumin soln. H. G. W.

Action of Normal and Immune Antibodies in Artificial Anaphylatoxin Formation. C. Morrischi and C. Vallardi. Inst. Gewerbekrankh., Milan. Z. Immunität., 11, 31-57.—Complement is indispensable for anaphylatoxin formation, but authors' results are inconclusive as to the role of normal amboceptor. Apparently very minute amts. of the normal amboceptor suffice for maximal formation of anaphylatoxin, while relatively large amts. of complement are necessary. Specific adsorption of fresh guinea-pig serum with typhoid, tubercle and cholera organisms, does not reduce the power of these sera to produce anaphylatoxin in vitro; and usually the toxigenic activity of the bacteria is not reduced. Very irregular results obtained with bacteria sensitized with immune anti-sera, as regards amt. of anaphylatoxin formed when acted upon by fresh serum, when contrasted with results obtained with non-sensitized bacteria. In no case, however, could the influence of the immune antibody be excluded.

H. G. W.

Complement Fixation with Strains of Typhoid, Paratyphoid and Allied Organisms. H. R. DEAN. Lister Inst., London. Z. Immunitat., 11, 58-85.—The complement fixation reaction furnishes an extremely delicate and specific means of separating and identifying closely allied races of bacteria, but careful quantitative work is necessary. Such closely related bacteria as paratyphosus A and B can be distinguished. The group antibodies can be removed from a serum by absorption, leaving the specific

antibodies. Excess of either the antiserum or of the bacterial extract has an unfavorable influence upon the binding of complement.

H. G. W.

The Biologic Function of some Salts of Alkali Metals and Alkali Earths. L. The Manifestations of Hemolysis. II. The Activities of Certain Pathogenic Bacteria. E. CALCATERRA. Maragliano's Inst., Genoa. Ann. inst. Maragliano, 4, 169-203; through Z. Immunitat., Ref., 4, 386.—The different salts investigated (NaCl, CaCl, K,SO4, MgSO4) have unequal hemolytic action in equimol. soln. with NaCl. The effects were dissimilar with both complement and amboceptor. In general it seemed that in the hemolytic phenomena the activity of the specific biological substances with high mol. comp. varies with the condition of chem. equil. between the different ions, and with the particular physical conditions, which result from the varying conc. of the salt molecules, as well as upon the variations in the colloid medium and the resistance of the red blood corpuscles. The second part is a report on the action of the same salts upon the B. pyocyaneus, a diplococcus, a putrifying organism, and typhoid and diphtheria bacilli; CaCl, had the most marked influence upon the growth of these bacteria, K.SO, and MgSO, following in order. The production of pigment by pyocyaneus was increased by MgSO4 and reduced by CaCl4. The diphtheria toxin formed on bouillon containing CaCl, was much less toxic than that from cultures with the three other salts. The agglutination of typhoid bacilli was delayed in cultures from bouillon containing these salts, most by NaCl. H. G. W.

Lecithin and Cultures of B. diphtheriae. E. CALCATERRA. Ann. inst. Maragliano, 4, 233-57; through Z. Immunität., Ref., 4, 387.—Small amts. of lecithin favor the growth of B. diphtheriae; large amts. decrease the virulence, growth, and toxicity of cultures.

H. G. W.

# 12. FOODS.

#### W. D. BIGELOW.

Hints for Food Manufacturers. Anon. Pure Products, 7, 479-80.—A brief description of the method of preparing several com. articles. W. D. Bigglow.

Sausage. A. REINSCH. Z. Nahr. Genussm., 22, 66.—Only rarely is a binding material necessary in making sausage. Starch (not over 2%) is the only binding material permitted.

D. S. Pratt.

Clams. P. BUTTENBERG. Staat. Hyg. Inst., Hamburg. Z. Nahr. Genussm., 22, 81-8.—A brief discussion of the habitat and use as food material of various species of clams. Bacteriological exam. showed that they are apt. to be infected with members of the colon group of organisms. Analysis of clams shows a resemblance to that of oysters.

D. S. Pratt.

Determination of Lecithin. C. VIRCHOW. Chem. Ztg., 35, 913-4.—The modified method finally adopted by V. is as follows: Ext. 1 g. of the substance 3 times with 10 cc. of b. abs. EtO, filter through a thick filter into a tared flask, washing with EtOH till the total vol. is 60 cc. Distil off the EtOH, treat with 10 cc. abs. Et<sub>2</sub>O, filter through asbestos, and wash with Et<sub>2</sub>O. Evap. off the solvent and weigh the residue. Oxidize the latter with fuming HNO<sub>2</sub>, pour into a Pt cruc., evap. to dryness, fuse with Na<sub>2</sub>CO<sub>2</sub> + KNO<sub>2</sub>, and finally det. P<sub>2</sub>O<sub>2</sub> as Mg<sub>2</sub>P<sub>2</sub>O<sub>7</sub>, from which the lecithin content may be calc.

WILLIAM BLUM.

Fancy Cheeses for the Farm and Factory. C. A. Publow. Cornell Univ. Agr. Expt. Sta., Bull. 270.—Methods of manuf. and comp. of various cheeses.

S. C. CLARK.

Summary of East Hungarian Sour Creams. JOSEF ADORJAN. Agri. Chem.

Foods. 3859

G. A. Roush.

Expt. Sta., Debuczin. Z. Nahr. Genussm., 22, 297-200.—Analyses of 36 samples of East Hungarian sour creams show an av. fat content of 20%, seldom less than 15%.

H. S. Balley.

The Composition of the Milk of some Breeds of Indian Cows and Buffaloes and its Variations. I. The Milk of some Breeds of Indian Cows. A. A. MEGGITT AND HAROLD H. MANN. Agr. Inst., Pusa. Mem. Dept. Agr. India (Chem. Ser.), 11, 1.— The evening milk is generally richer than the morning milk, the difference usually being less marked during the rainy season. The av. fat content of the "Gir" breed is: morning milk, 5.2%; evening milk, 6.2%. Of the "Sind" breed, 6.0 and 6.3%, resp. The comp. remains quite const. regardless of the yield, rizing slightly in fat content toward the end of the period of lactation, this being more pronounced in the "Sind" cows than in the "Gir." The yield rizes to a max. almost at once, and remains almost const. for about  $\frac{3}{16}$  of the lactation period, followed by a steady decline. Some

Sterilac. An Apparatus for Aseptic Milking, Preserving and for the Preparation of Modified Milk. Siegeried Weiss and Victor Brudny. Lab. K. K. Hochschule f. Bodenkultur. Arch. Kinderh., 56, 129-49.—The necessity of using ice in the app. "Sterilac" (S. Weiss, Wien. med. Wochenschr., 1908, No. 2), might prevent its use in places where ice is not obtainable. The author, therefore, tried out various cheap freezing mixts. without ice. The use of a mixt. of 300 g. KNO<sub>2</sub>, 300 g. (NH<sub>4</sub>)Cl, and 300 g. Na<sub>2</sub>SO<sub>4</sub> dissolved in 1 l. water (reducing the temp. from 10° to —15.5°) in the app. permitted the preservation of 1 l. milk below 10° for 20 hrs. The author describes the use of the app. in the prep. of modified milk.

Walter A. Jacobs.

tests showed a fat content as high as 0.0%.

Reduction of Nitrates through the System; Perhydridase-Aldehydes-Water. A. BACH. Biochem. Z., 33, 282-90; also J. Soc. Chem. Ind., 30, 917.—A continuation of C. A., 5, 2258. Measured quantities of milk, NaNO, soln, and the aldehyde soln, were mixed and after making up to 100 cc., shaken in a thermostat. From time to time 1-2 cc. samples were drawn into a 50 cc. flask containing 1 cc. of 1% basic Pb(OAc), soln., the flask filled to the mark and the soln. filtered. This gives a clear filtrate which is readily tested for N<sub>2</sub>O<sub>2</sub> content by the Slesvay-Lunge colorimetric method. Working with varying amts. of acetate and formaldehyde, cooked and raw milks, and NaNO, solns. at diff. temps. B. concludes that: (1) Raw milk accelerates the reduction of nitrates by aldehyde. (2) Boiled milk with aldehyde or fresh milk alone has no effect on nitrates. (3) The rate of reduction increases with increase of aldehyde or nitrate content but not proportionally. (4) With const. aldehyde and NO<sub>a</sub> content the rate of reduction is, in the early stages, proportional to the enzyme content but not in the later phases. (5) A decomp. of the nitrite takes place coincident with the NO<sub>2</sub> reduction, and both reactions increase with rise in temp. up to 60-70°. (6) The AcOH formed has no effect on the reaction, as the rate is the same in NaHCO. as acid solns. (7) In a mixt. of milk, CH<sub>2</sub>CHO and NaNO, held at 50° there is a very slight decrease in the NO<sub>2</sub>. (8) HCHO gives less favorable results than CH<sub>2</sub>CHO. (9) If calf's liver be extracted with a 20% soln. NaHCO, and 1% NaF a perhydridase similar to that of milk is obtained. H. S. BAILEY.

Fat Content of Milk from Cows of the Normandy Breed. (Average Butter Production.) CH. BRIOUX. Agron. Sta., Lower Seine. Ann. jals., 4, 470-5; cf. C. A., 5, 728.—Tables showing av. milk production, a. m., noon, and p. m., and av. quantity of butter for each month of 1910 from 150 cows are given. The milk from the noon milking is richest, but the production highest in the morning. For the whole 24 hr. periods the av. fat in 1909 was 39.1 g. per l., in 1910, 39.4, showing slight improvement in the general dairy conditions.

H. S. Bailey.

The Difference between Fruit Essences and Artificial Fruit Esters. Weeners. Schweis. Wockschr., 49, 385-6.—A number of natural essences and artificial substitutes were examd. comparatively by detg. the d., d. of distillate, ester content, and volatil acids in the distillate. In an artificial strawberry essence, valeric, butyric and acetic acids were identified while the natural essence showed propionic and acetic acids.

H. C. FULLER.

Canned Corn. A. McGILL. Inland Rev. Dept., Ottawa, Canada, Bull. 226.—Results of the exam. of 146 samples of Canadian canned corn are given. The wt. of contents varied from about 20 to 22 oz. Decided amts. of Na<sub>p</sub>SO<sub>2</sub> were found in 46 samples and traces in 27 others. In 29 samples saccharin was found.

W. D. BIGELOW.

Pleurotus cretaceus, an Indian Edible Mushroom. David Hooper. Calcutta. Chem. News, 104, 145.—The fungus is sold in a dry state. Before cooking it is soaked in fresh water for about 8 hrs., when it swells and becomes pulpy. It is highly prized. The % analysis of the dried product is as follows: H<sub>2</sub>O 12.25, N 3.40, crude fiber 305, ash 8.75, P<sub>2</sub>O<sub>4</sub> 1.85.

W. D. BIGELOW.

Composition of Sauerkraut. E. FEDER. Z. Nahr. Genussm., 22, 295-6.—Sauerkraut was dried and extracted with 90% alc. giving crystals, m. 166.5°, optically inactive in soln., dextro after addition of 5% borax. Acetate: m. 118.5°, sapon. no. 771, identified as mannitol, and comprizes 10% of the dry residue.

D. S. PRATT.

The Determination of Benzoic Acid. Otto Folin and Fred F. Flanders. J. Am. Chem. Soc., 33, 1622-6.—The proposed method as applied to catchup is as follows: 25 g. are weighed out, 2 cc. conc. HNO<sub>2</sub> added and the mixt. well stirred. Several small portions, about 0.2-0.3 g. in all, of NaNO<sub>2</sub> are added, stirring well after each addition. Without filtering, the bleached mass is rinsed into a 500 cc. separatory funnel by means of 200 cc. of satd. (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> soln. Extractions are made with washed CHCl<sub>2</sub>, using 50, 35, 25, 25 and 25 cc., resp. The combined ext. is then shaken with two 200 cc. portions of satd. NaCl soln. containing 3 cc. 10% HCl. This removes AcOH, glycolic and lactic acids if these are present. The washed ext. is then titrated with standard Na alcoholates (2.3 g. Na in 1 l. abs. alc.), using phenolph. The Na alcoholate soln. should be standardized against pure BzOH in soln. in 165 cc. CHCl<sub>2</sub>. The method is rapid, easy and economical, and the results reported on catchup agree very closely with the amts. of BzONa added.

P. B. Dunbar.

Detection of Benzoic Acid in Food. Ep. Polenske. Arb. Kois. Gesundh., 38, 149-54.—P. finds the method of Fischer and Gruener to be at least as sensitive as that of Mohler when properly conducted. He suggests the following details for its manipulation: The substance is placed in a Ag crucible with coarsely powdered KOH. The crucible rests with its bottom about 2½ cm. above a burner with a flame 3 cm. high. The contents are stirred with a Pt wire and should be fused from 35 to 45 sec.; the heating is continued from 2 to 21/2 mins. longer. The fusion is then tested for salicylic acid in the usual manner. P. detd. the amt. of BzOH in cranberries by the following method: 50 g. of the material ground to a pulp, were mixed with 300 cc. of 96% alc. in a 500 cc. flask. A reflux condenser was attached and the mixt. heated at 70° for 1 hr. It was then cooled to 40°, NaOH added till a green ppt. forms and the mixt. cooled to 15°, filtered through a porous plate and the residue washed 5 times with 200 cc. of 96% alc. Alc. is removed from the filtrate by evapn. and the residue, measuring about 40 cc., is strongly acidified with H<sub>2</sub>SO<sub>4</sub> (1:3) and filtered into a 200 cc. filter, the residue and filtrate being washed until the filtrate is colorless. The filter is shaken for 1 min. with 50 cc. of ether and 50 cc. of petroleum ether, resp. The ether soln, is sepd, and the BzOH extracted from it by shaking with 10 cc. of 5% NaOH. The ether mixt. is again used for a second extraction of the original liquor Foods. 3861

and again washed with the NaOH soln., this operation being conducted 5 times in all. The alk. soln. containing BzOH is now transferred to a porcelain dish, heated on a water bath, and treated drop by drop with a satd. soln. of KMnO<sub>4</sub> until a red color remains for several min. The excess of KMnO<sub>4</sub> is then removed by a satd. soln. of Na<sub>2</sub>SO<sub>4</sub>, acidified with H<sub>2</sub>SO<sub>4</sub> and the addition of Na<sub>2</sub>SO<sub>5</sub> continued until the sepd. MnO<sub>5</sub> is dissolved. BzOH is then sepd. by 4 extractions with 15 cc. each of ether, the solvent evapd. and the BzOH finally sepd. by subliming through a filter paper and detd. by titration.

W. D. Bigelow.

Molasses Fodders. CROCHETTELLE AND MILON. Sta. Agron. Somme. Rev. chim. ind., 22, 279-85.—Very nutritive fodders are obtained by adding molasses to substances having food value of themselves such as brewers' malt, bean-, rice-, and oat-meals, grape and apple pomaces, and casein from dairy waters. Less nutritive fodders are obtained by using coffee chaff, peanut and cocoa shells, and peat. The digestibility of these may be increased by the action of the molasses. Peat combines with Na and K salts and does not absorb H<sub>2</sub>O. The analysis of typical molasses fodders gives an av. of about 20% H<sub>2</sub>O; 20 to 30% sugar; and N varying from 10 to 20%. During storage there is an increase in H<sub>2</sub>O, an inversion and loss of sugar, and invasion by organisms. For a routine study of fodders the sugar before and after inversion is detd. as a measure of molasses, and microscopic exam. is made to reveal the nature and relative amts. of absorbents. In prep. of the sugar soln. for analysis, the extraction of material with hot H<sub>2</sub>O, addition of alc. and basic Pb acetate, and filtration is used, allowance being made for the vol. of insol. matter.

E. L. P. TREUTHARDT.

The Determination of Fat in Feeding Stuffs. B. SCHULZE, O. BIALON, F. WERNER, R. GORKOW AND G. KLOSE. Landw. Vers. Stat., 75, 185-230.—After an exhaustive research the authors come to the following conclusions: (1) The substance to be extracted must pass through a 1 mm. sieve; (2) the extraction must be made with anhydrous ether; (3) the drying of the substance should be conducted with great care. Peanut, cottonseed, sesame seed, hemp and rape meals can be dryed in an ordinary oven. Both the substance and the fat from it should be dried for 2 hrs. Flaxseed meal and the fat from it should be dried in O-free gas for 2 hrs. Coconut, palm, rice and millet meals and fermented maize residue should be dried in an ordinary oven 1 hr. and the ether ext. 2 hrs. The short time of drying is an attempted compensation for H<sub>2</sub>O and volatil fats. The temp. for drying should be from 98 to 100°.

E. C. LATHROP.

Exact Determination of Nicotine in Tobacco and in the Green Plants of Nicotiana tabacum. R. Kissling. Schweiz Wochschr., 49, 387; cf. C. A., 5, 1472, 1807.— The author claims that Mellet's method is really his, deteriorated for the following reasons: The use of CaO instead of NaOH, producing a resinate which renders difficult a complete extraction of the nicotine, the extraction from aq. soln. instead of a dry mixt., the filtration of the Et<sub>2</sub>O soln. and the loss of nicotine. H. C. Fuller.

Exact Determination of Nicotine in Tobacco and in the Green Plants of Nicotiana tabacum. R. Mellet. Schweis Wochschr., 49, 387-8.—In reply to above criticisms the author states that Kissling's methods are not applicable to the green plant for which his was intended exclusively, and grants that it is inferior to others for dry tobacco extracts. Higher results are obtained with CaO than with NaOH. H. C. F.

Recin in Feeding Stuffs (MOOSER). 11b.

BAUER, H.: Hahrungsmittelchemisches Praktikum. Stuttgart: 8°, 266 pp., 7 M.

- GRIFFITHS, W.: The Principal Starches Used as Food. 2nd Ed. Circnester: Bailey & Woods, 6 s.
- LEACH, A. E.: Food Inspection and Analysis. 2nd Ed. New York: John Wiley & Sons. 954 pp., \$7.50.
- Lenzen, H.: Ueber die Bedeutung und den praktischen Wert der gebräuchlichsten Untersuchungsmethoden der Milch. Leipzig: 8°, 1.20 M.
- RIDEAL, S.: Disinfection and the Preservation of Food. 3rd Ed. London: 494 pp., \$4.50.
  - SÉRAND: Le pain. 2nd Ed. Paris: 8°, 208 pp.
- U. S., 1,001,044, Aug. 22. J. H. Kellogg, Battle Creek, Mich. Food compound, comprizing corn- or nut-oil ½ lb., gluten 3 lbs., legumes, e. g., peas, beans or lentils 1 lb., and yeast ext. 2-4 g. per lb. or crude yeast mixt. 1-2 oz., combined with egg and salt and cooked into a homogeneous mixt. of fibrous consistency and meat-like flavor.
- U. S., 1,001,045, Aug. 22. *Idem*. Food compound, comprizing gluten 2-3 lbs., nuts (preferably dry peanut meal) 1 lb., and crude yeast or conc. yeast ext., cooked into a homogeneous mixt. of fibrous consistency and meat-like flavor.
- U. S., 1,001,149, Aug. 22. *Idem*. Food compound, comprizing corn- or nutoil <sup>1</sup>/<sub>4</sub> lb., gluten 1 lb., potato meal 1 lb., conc. yeast ext. or crude yeast and egg cooked into a homogeneous mixt. of fibrous texture and meat-like flavor.
- U. S., 1,001,150, Aug. 22. *Idem*. Food compound, comprizing casein 3, gluten (wet) 3, corn- or nut-oil 2 parts, and crude yeast or yeast ext., 2 g. to 2 oz. per lb., cooked into a homogeneous mixt. of meat-like flavor.
- U. S., 1,001,253, Aug. 22. J. J. A. CROLBOIS, Paris, France. Making a cattle food by spraying vegetable matter (e. g., beet root pulp) in silos with a culture liquid containing a lactic ferment acclimated to the medium in which it is to develop, to increase the stability, digestibility and assimilability of the food.
- U. S., 1,001,375, Aug. 22. T. EDWARDS, Washington, D. C. Preparing oysters, clams or other shell fish for food by grinding them and placing them in a heated receptacle, agitating the mass to break it up into small particles and at the same time forcing heated air under pressure therethrough to expel the moisture and reduce the mass to a mushy consistency, and finally evapg. and pulverizing.
  - U. S., 1,001,517, Aug. 22. O. EICK, Baltimore, Md. Pasteurizing apparatus.
- U. S., 1,001,554, Aug. 22. E. G. RESSENCOURT, St. Louis, Mo. Making a fruit extract by crushing the fruit with an equal quantity of rock candy, adding 1/2 by vol. of cologne spirit, allowing it to stand, distilling the mixt. at 93° until the vol. is reduced 1/2, straining the residue and mixing it with the distillate.
- U. S., 1,001,555, Aug. 22. *Idem*. Making peach extract by crushing a mixt. of sugar and the whole fruit, adding cologne spirit, muscatel wine and peach spirit and distilling at about 93° until the vol. is reduced about ½, straining the residue and adding it to the distillate together with the liquid resulting from crushing peach skins and treating them with cologne spirit.
- U. S., 1,001,556, Aug. 22. Idem. Making strawberry or raspberry extract by crushing a mixt. of rock candy and the whole fruit, adding cologne spirit, angelica wine and grape spirit, distilling at about 93° until the vol. is reduced about ½, straining the residue and adding it to the distillate.
- U. S., 1,002,020, Aug. 29. W. A. D. Allport and T. J. W. C. Davenport, Roseau, Dominica, British West Indies. Machine for extracting essential oils from citrous fruits, comprizing a weighted traveling pressing device and a fixed puncturing device

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arranged parallel thereto, a feeder for feeding the fruits 1 by 1 between the devices at 1 end to be rolled along between them and punctured and pressed to expel the oils contained in their skins, a chute at the other end of the devices for receiving the fruits, and water jets at the chute to wash off the oil adhering to the surface of the fruits during their passage down the chute.

- U. S., 1,002,106, Aug. 29. S. WITHERELL, Ottumwa, Iowa. Milk tester having a milk receptacle with end chambers adapted, resp., for the reception of milk and butterfat when sepd., sealing devices for the chambers, and a pivotly mounted balance for indicating the shifting of the center of gravity of the receptacle.
- U. S., 1,002,137, Aug. 29. L. B. G. CARRERAS, Morovis, Porto Rico. Composition for making coffee rapidly, consisting of dry coffee ext. 60, sugar of milk 270, NaHCO<sub>3.3.6</sub>, citric acid 2.4, and gum arabic 10 parts.
- U. S., 1,002,496, Sept. 5. C. H. CAMPBELL, New York, N. Y. Making dry milk by partially concg. milk, spreading it in the form of successive films upon one another and drying the films sufficiently to form a thick body of solidified milk, allowing this body to set or stiffen and removing from it sep. small portions by cutting, which are adapted to dry readily and crumble to a light uncompacted mass.
- U. S., 1,002,499, Sept. 5. R. M. CAUFFMAN, Centerville, Mich. Apparatus for pasteurizing milk.
- U. S., 1,002,506, Sept. 5. C. Ellis, Montclair, N. J. Seasoning composition, comprizing substantially unoxidized and undecomposed, vacuum-dried onion juice intimately incorporated with NaCl and in dry pulverulent form.
- U. S., 1,002,707, Sept. 5. I. I. Kononoff, Astrakhan, Russia. Preserving fish by injecting into them, immediately after killing and before the circulation of the blood ceases, a pure NaCl soln., at one or more places under the scales of the fish.
- U. S., 1,002,990, Sept. 12. C. HERENDEEN, Chicago, Ill. Making flour or meal from grits, meal, etc., by injecting steam into the material for a sufficient time to disrupt the starch cells, passing the product through blasts of air to dry it and then passing it into grinders and reducing it to the desired fineness. The product is stated to be capable of absorbing 2-6 times its wt. of H<sub>2</sub>O and is intended to be mixed with other less absorbent flour.
- U. S., 1,003,151, Sept. 12. F. Quade, Berlin, Ger. Assignor to J. A. Wülfing, same place. Making a non-hygroscopic albuminous composition for use as a food and tonic, by preparing the raw material by dissolving Na metaphosphate in H<sub>2</sub>O, adding glycerol, heating the mixt. in vacuo to evap. the H<sub>2</sub>O and sepg. the phosphate in a divided state in the glycerol, heating to 145° to dissolve and esterify the phosphate, adding H<sub>2</sub>O and allowing the mixt. to cool, introducing it into a menstruum containing casein-Na containing 1% Na while agitating the entire mixt., digesting the latter, sepg. the combination of casein-Na with alkali glycerophosphate by filtration from the mixt., and finally drying.
- U. S., 1,003,320, Sept. 12. A. BAUMANN, New York, N. Y. Pickling green cucumbers by packing them in a vessel, subjecting them to the action of a brine containing 20-32% NaCl with 2-8 oz. HCl and 2-8 oz. Na benzoate to every 20 gals. of brine, allowing the brine to penetrate the cucumbers, removing a portion of the brine and finally closing the vessel.
- U. S., 1,003,525, Sept. 19. W. VON SKORZEWSKI, Schloss Lubostron, near Labischin, Ger. Producing durable fodder from fresh vegetable matter, e. g., turnip and beet root, leaves, grass, etc., by comminuting and mixing the material to a pasty consistency; molding without pressure and then drying at atm. temp. and pressure. The material is preferably also steamed below:

U. S., 1,003,826, Sept. 19. C. W. TICKNOR, Mount Kisco, N. Y. Device for aerating, cooling and cleaning milk.

Brit., 20,136, Aug. 29, 1910. E. D. Houston, 432 Part Ave., Worcester, Mass. Mfg. a seasoning by peeling or cutting up a vegetable of such a nature that its exposed surfaces will be moist, mixing therewith a quantity of NaCl, grinding the mixt. to a pulp, and evapg. the same until the mixt. assumes a flaky form.

### 13. NUTRITION.

wm. j. gies. NORMAL.

Experiments on the Absorption of Gelatin in the Small Intestin. D. Minami. Chem. Abteil. physiol. Instit. Breslau. Biochem. Z., 34, 261-2.—Expts. with dogs showed that a pure H<sub>2</sub>O soln. of gelatin was not absorbed by the small intestin. Gelatin previously digested with pepsin-HCl was absorbed. After digestion with pancreas ext. the absorption appeared to take place more quickly. The statement made by E. S. London that after feeding gelatin the absorption takes place chiefly in the lower part of the small intestin could not be confirmed.

F. J. BIRCHARD.

Protein Metabolism. II. FRANZ FRANK AND ALFRED SCHITTENHELM. Lab. d. Erlanger med. Klinik. Z. physiol. Chem., 73, 157-75; cf. C. A., 5, 1307.—The N balance of 2 dogs was detd. daily for 6 and 41/2 months, respectively. The food in each case contained from day to day the same amt. of fat, starch, sugar and N, the N being little above the hunger value. As sources of N the following preps, were employed: Egg albumin; the dried and powdered flesh of ox, horse, dog and fish; the products of digestion of skimmed-milk powder, casein, egg albumin and serum albumin; and 2 com. preps., "ereption" from beef, and "riba" proteose from fish. The digested products were prepared by treatment for 1 week with pepsin-HCl, 3 weeks with pancreatin, and I week with erepsin; they gave no biuret reaction. The sep. diet periods lasted 7-26 days. The results of each expt. were the same. The animals maintained their health and wt. The N balance of the one was, except in the riba period, continuously positive, although for 2 months the diet was strictly biuret-free. The other suffered only at first a slight loss of N. The value in protein metabolism of the different nitrogenous materials was practically the same. The utilization of the biuretfree mixts. stood little behind that of the proteins from which they were derived; thus the daily N excretion of egg-albumin was 0.86 g., on its digestion product 0.93 g. The preps. utilized best of all were egg albumin and casein; the least satisfactory was riba; flesh occupied an intermediate position. Dog flesh showed no special suitability. A. HUNTER.

Investigations on the Acidity of the Urin with Studies on Infants. O. Lade. Akad. Klinik f. Kinderh., Düsseldorf. Arch. Kinderh., 55, 161-99.—D. sought to det. any differences to be found in the acidity of the urin of breast babies and bottle babies and whether gross alimentary influences can affect this acidity. In both cases the acidity is little influenced by the amt. of food assimilated, but that of the breast child is markedly lower (0.3-0.4 cc. 0.1 N NaOH per 10 cc. urin; for bottle baby 1.2-2.9 cc.). A greater daily acidity corresponds in general to a larger quantity of urin. The P curve in both cases parallels the acidity curve. In the breast babe the P values are low and can affect the acidity in small measure only. In bottle babies it is occasionally so high as to more than correspond to the acidity. P<sub>9</sub>O<sub>6</sub> may be present then as primary phosphate. The N curve runs in the same direction as the acidity curve.

Protein Addition in the Fattening of Sheep. T. Preiffer and K. Friske. Londw. Vers. Sta., 74, 409-65.—By feeding sheep in 10-12-day periods for 110-117 days with different feeding ratios, and studying the N balance and the flesh and fat of the slaughtered animal, the authors come to the following conclusions: A marked addition of N in the form of flesh, in the true sense of the word, takes place during the fattening of sheep. The N addition reaches a max. and then remains const. until the animals are put on a poorer diet. A diminution of the protein in the feeding ration does not influence the duration of flesh production. The study of the metabolic assimilation indicated a higher N addition than slaughter expts. By both methods the results obtained from the feeding of protein-poor rations showed good agreement.

E. C. LATHROP.

Further Investigations on the Value of Ammonium Acetate and Asparagine for the Support of Life and for Milk Secretion. A. MORGEN, C. BERGER AND F. WEST-HAUSER. Landw. Vers. Stat., 75, 265-320.—Protein food gave the largest amt. of milk; by replacing a considerable amt. of protein with NH<sub>4</sub>OAc or asparagine the secretion of milk was retarded about 25%; by replacing the protein with carbohydrates it was retarded still more (about 36%). NH<sub>4</sub>OAc and asparagine acted alike in all respects and seemed to have no influence on body wt. A formation of the so-called bacterial protein from NH<sub>4</sub>OAc or asparagine cannot take place because by the feeding of these substances no greater amt. of protein was found in the feees than by the feeding of carbohydrates. The N of the NH<sub>4</sub>OAc or asparagine in a protein-poor dlet which contained sufficient starch, had a value in all expts. of 32.2%, not only for the maintenance of life but for the production of milk, wool, or flesh. The amt. of metabolic assimilable products with a protein diet, or its substitution by NH<sub>4</sub>OAc or asparagine, was normal; by the substitution of carbohydrates it was somewhat higher.

E. C. LATHROP.

The Value of a Flesh Food for Herbivorous Animals. F. Honcamp, B. Gechwender and D. Engerding. Landw. Vers. Stat., 75, 161-84.—The authors experimenting with sheep and replacing a diet of clover hay with different kinds of flesh foods, herring meal, whale meal, "kadavermehl" and dried blood, show that the animal is able to make good use of the protein and fat which they contain, and that they may be made use of as a cattle food.

E. C. Lathrop.

The Influence of Oxygen Inhalations on the Lactic Acid Produced during Hard Work. ISRAEL FELDMAN AND LEONARD HILL. London Hospital Med. Coll. J. Physiol., 42, 439-43.—Assuming that the adequate stimulus of the respiratory center is the acid or H-ion content of the blood, O inhalations will act by diminishing the lactic acid in the muscles and the acid conc. of the blood (C. A., 4, 2691). The expts. showed that after a short period of exertion almost no lactic acid appears in the urin if O is inhaled, and a distinct amt. if air is breathed. It is concluded that the increased production of lactic acid by the muscles is due to O want. Thus want of O by increasing the acid content of the muscles and blood may stimulate the respiratory center.

I. F. LYMAN.

The Growth of the Body as Influenced by Feeding Homologous and Heterologous Body Proteins. Edward Babar, Prag. Zentr. Physiol., 25, 437-41.—A comparative study on frogs, Rana fusca, of the influence of feeding them (a) frog's muscle, (b) lean beef, (c) crab meat, and (d) muscle of anodonta. The frogs thrived best (increase of wt.) on (d). The length of the digestive tract became greatest on feeding (c). Taking the protein content of the foods into account, (d) was best adapted for producing the body substance of the frog, then follow (a), (b) and (c). G. M. MEYER.

Absorption and Elimination of Starch Grains. FRITZ VERZAR. Physiol. Chem. Lab., Univ. Budapest. Biochem. Z., 34, 86.—V. corroborates the results of R. Hirsch,

who showed that starch grains may be absorbed by the intestinal epithelium and eliminated unchanged in the urin. These expts. were carried out on men, dogs, rabbits and rats.

G. M. MEYER.

Relation of the Melting Point of Fats to their Rate of Disappearance from the Stomach. Franz Tangl. and Alexander Erdelyi. Physiol. chem. lab., Univ. Budapest. Biochem. Z., 34, 94-110.—Higher m. fats disappear less rapidly from the stomach than those with a lower m. p. The rate of disappearance depends on the viscosity of the fats; those with a higher viscosity passing out more slowly. The relative rate of disappearance is the same if the fats are administered in the form of an emulsion.

G. M. Meyer.

Fat Digestion. STEPHAN VON PESTHY. Physiol. chem. lab., Univ. Budapest. Biochem. Z., 34, 147-75.—The extent of fat digestion may be followed accurately by glycerol detns. The fatty acid detn. is only of importance if the fat digestion is not accompanied by other digestive processes. Fat digestion in the stomach is due to a sp. enzyme secreted by the stomach and not regurgitated from the intestins. Both fat and glycerol may be detected in the stomach contents after fat digestion but not in the ratio as they exist in the fat mol.

G. M. MEYER.

#### ABNORMAL.

The Influence of Urea on the Blood and Milk of Nursing Woman. Sr. Engr. And H. Murschhauser. Akad. Kinderklinik, Düsseldorf. Z. physiol. Chem., 73, 131-7.—Three expts. were made on 2 young primiparae in the 5th month of lactation. Daily doses of 15 g. urea were given for periods of 4, 3, and 3 days. Total N and "rest-N" of blood serum and milk were detd. before, during and after each period. In the 1st expt. "rest-N" in 100 cc. milk rose from 0.0386 to 0.0498 g., or from 24.77 to 28.49% of the total N; in 100 cc. serum "rest-N" rose from 0.0449 to 0.0500 g., or from 3.33 to 3.63% of total N. The increase in the milk corresponded to an extra secretion of at least 0.5 g. urea daily. In the other expts. similar results were obtained; in each case an abs. as well as relative increase of "rest-N" in milk and serum. The human mammary gland is therefore capable under circumstances of acting as an excretory organ for urea.

A. Hunter.

Calcium Metabolism in a Case of Infantile Tetany. Howard D. HASKINS AND H. J. GERSTENBERGER. Western Reserve Univ., Cleveland. J. Exp. Med., 13, 314-8.—Contrary to the results of others with dogs, in the human case observed by the authors, administration of Ca salts and parathyroid did not favor Ca retention.

WALTER A. JACOBS.

Changes in Nitrogenous Metabolism after Parathyroidectomy. JEAN V. COOKE. New York. J. Exp. Med., 13, 439-54. — After complete parathyroidectomy (in dogs) a marked and constant increase in the urinary N and NH<sub>2</sub> occurred with an increased NH<sub>2</sub> ratio. The creatinine and amino acids remained practically unaltered with possibly a slight increase in the undetd. N. The increase in the NH<sub>3</sub> and the undetd. N occurred at the expense of the urea fraction. In several of the animals lactic acid appeared in the urin coincident with the tetany and with the increased elimination of N and NH<sub>2</sub>. C. connects this formation of lactic acid with diminished oxidation. The increase in NH<sub>2</sub> and Mg of the urin may be the result of over-production of these bases to offset the excessive acid. This removal of bases may by altering the salt balance of the nerve cells produce a hyperexcitability of the central nervous system resulting in tetany.

Walter A. Jacobs.

The Mechanism of Chlorine Retention in Pneumonia. FLORENTIN MEDIGRECEANU. New York. J. Exp. Med., 14, 289-97.—The Cl output is very much diminished during the process of experimental pneumonia in dogs. The quantity of Cl retained in

the cases with well developed exudates is much larger than the exudate could account for. Turpentine pleurisy in dogs also is associated with a retention of Cl, but this is not so marked as in pneumonia. There is no direct quant. relation between the water output and the Cl excretion. The rabbits with pneumococcal septicemia did not retain Cl in any phase of the disease.

Walter A. Jacobs.

Experimental Obstructive Jaundice. John H. King, J. E. Bigelow and L. Pearce. Johns Hopkins Med. School, Baltimore. J. Exp. Med., 14, 159-78.—In obstructive jaundice, produced in dogs by ligation of the common duct, there is a loss of Ca by the jaundiced animal. The Ca is given up mainly by the bone, to neutralize the toxic bile pigments circulating in the blood and tissues. This neutralization affords protection to the body, but may lead to secondary disturbances, such as bradycardia and delay in the coagulation time. These two latter signs of biliary toxemia are due, perhaps, in some measure to the removal of Ca. The increased elimination of Ca occurs chiefly in the feces, and only to a small extent through the kidneys. Walter A. Jacobs.

The Influence of Lime- and Phosphate-poor Feed on Milk Secretion. G. FINGER-LING. Landw. Vers. Stat., 75, 1-152.—Cows are fed rations containing different ratios of CaO and  $P_2O_4$  and the distribution of these compds. between the milk, animal, urin and feces are studied. If milk-giving animals are not fed the amts. of CaO and  $P_2O_4$  necessary for the maintenance of the milk, the other organs lose this amt. from their supply without the activity of the milk glands being injured. By a long period of feeding of rations poor in these 2 ingredients, the secretion of milk diminished and the milk is of poorer quality including the content of CaO and  $P_4O_4$ . If the feed is raised in % of lime and phosphates the organs quickly take up the amts. of these compds. they have lost and milk improves in comp. and quantity. The amts. of CaO and  $P_4O_4$  fed have only a weak influence on % comp. of the milk; in the milk ash the % of CaO and  $P_4O_4$  is raised quicker than it is lowered.

E. C. Lathrop.

Sex and Metabolism. R. A. Krause and W. Cramer. Proc. Physiol. Soc., 1911, 34-5; J. Physiol., 42.—Creatine is present in the urin of pregnant women, the max. amt. found being 0.15 g. per diem. Creatine is found in the urin of women living on a creatine and creatine-free diet immediately after menstruation. Creatinuria in normal women is associated with a relatively high NH<sub>2</sub> excretion. J. F. Lyman.

Resorption of Reserve Fat. Maurice Piettre. Compt. rend., 153, 487-90.

—Reserve fat resorption in conditions of cachexia takes place with extreme rapidity from all adipose depots simultaneously.

G. M. Meyer.

Metabolism of Starch Introduced Parenterally. F. VERZAR. Physiol. chem. Lab., Univ. Budapest. Biochem. Z., 34, 66-85.—When starch is rapidly injected intravenously it soon appears in the urin. If the injection is done slowly none is eliminated. That the starch is oxidized was shown by an increase in CO<sub>2</sub> formation with a corresponding rise in the CO<sub>2</sub>/O<sub>3</sub> quotient. The intravenously injected starch is hydrolyzed in the blood prior to its oxidation. Starch emulsion can be injected into dogs with extirpated pancreas in much larger amts. than into normal animals. This is due to the increase of the blood diastase in the former animals. G. M. MEYER.

Influence of Intravenous Blood Transfusion on the Metabolism of Matter and Rnergy. P. Hari. Physiol. Chem. Lab., Univ. Budapest. Biochem. Z., 34, 111-46.

—Expts. were performed on dogs. Blood transfusion was followed by an increase in urinary N. Fat metabolism was lowered. On starving animals there is water retention which could not be detected on well fed animals. There is an increase in heat production due to an increased heart action.

G. M. MEYER.

Calcium Content of Woman's Milk. ARNALDO RAMACCI. Kinderklin., Parma. La pediat., 18, 665-9; through Zentr. Biochem. Biophys., 11, 882.—By Neumann's

method the CaO content of 68 samples of woman's milk was found to vary between 0.0291 and 0.2791 g. with an av. of 0.1024 g. As has been observed before, infants suffering from rachitis were found to be nourished by milk of low Ca content.

I. K. PHELPS.

Influence of Diminution of Oxidation Processes on Metabolism as Shown in the Urin. V. Scaffidi and V. Girone. Inst. allg. Pathol. Neapel. La sper., 64, 537-48; through Zentr. Biochem. Biothys., 11, 875.—The normal oxidation of the tissues in a dog was stopped, and during the interval the animal was kept under a glass bell jar into which a mixt. of air and CO<sub>2</sub> were driven. The analysis of the urin excreted for 24 hrs. showed that the excretion of urea and NH<sub>2</sub> had been lessened decidedly and the creatine somewhat by the inhibition of the processes of oxidation. The excretion of total N and S in the urin did not undergo any corresponding diminution, which leads to the conclusion that the cleavage of proteins is not influenced quantitatively. The decrease of NH<sub>2</sub> and urea are due rather to an incomplete cleavage of the intermediary products of the proteins.

## 14. WATER, SEWAGE AND SANITATION.

#### EDWARD BARTOW.

Amounts of Ammonia and Nitric Acid in Rainwater in Tonquin. M. AUFRAY. Bull. Économ., Hanoi-Haiphong, 12, 595-616; through J. Chem. Soc., 100, II, 224.— N as NH<sub>2</sub> and NO<sub>2</sub> was detd. in 436 samples of rain water collected in 1902-9 at Hanoi, a city in French Indo-China. Total N per acre per annum in rain ranged from 6.69 to 28.21 lbs., but the quantity is not proportional to amt. of pptn., though 84% of total N is contained in the 82% of the rain that falls from May to Oct.

R. B. DOLE.

Significance of Flora and Fauna in Maintaining the Purity of Natural Waters and How they are Affected by Domestic Sewage and Trade Wastes. E. Kuichling. Enq. News, 66, 246-50.—A translation of a paper written in 1909, by Dr. Maximilian Marsson. The plankton must be divided into 2 classes, food producers, and food consumers. The plants which assimilate inorganic matter and build up organic compds. by means of their chromophyll coloring matter belong to the first class; and all other organisms, such as microscopic protozoa, rotifera, etc., belong to the second class. The vegetable plankton varies with the seasons and with the character of the water. So-called "blooming" of rivers and lakes is usually produced by the Schizophyceae; the other group of algae have only a few representatives in fresh water. All of these plants dissociate O from H<sub>2</sub>CO<sub>2</sub>. The N food is obtained from nitrates and NH<sub>2</sub>. As many organisms of different kinds were found in winter as in summer. The variations of temp. had no effect, and the only other difference was in the mode of nutrition. Diatoms as well as green algae can not only absorb C compds, from dissolved organic matter, but also organic N. The direct absorption of dissolved organic matter by algae is of the utmost importance for the purification of streams by eliminating therefrom the sol. products of putrefaction. Molds or fungi rely for their food on the presence of other organisms, or of suitably prepared organic matter. They are either parasites or saprophytes. The bacteria (Schizomycetes) consume O. The green algae counteract the bacterial consumption of O. The bacteria liquefy the organic components of dead vegetable and animal matter and render them capable of being absorbed by living plants. The Beggiatoa can exist only in flowing water. Its rapid growth takes place only when H<sub>2</sub>S is present as a product of putrefaction which subsequently becomes oxidized into H<sub>2</sub>SO<sub>4</sub>. The various fungi serve as an index of the source and the degree of pollution. The number of species of animals found at the bottom of rivera

such as shell fish and aquatic snails contribute to the purification of waters by ingesting plankton and pseudo-plankton, as well as small masses of fungi. The quantity of putrescible matter must always be in proper proportion to the vol. of the stream if a nuisance is to be avoided. Certain trade wastes, such as tarry and alk. wastes, interfere seriously with the self-purification of rivers, inasmuch as they are harmful to aquatic animal and plant life.

A. Lederer.

The Geochemical Interpretation of Water Analyses. Chase Palmer. U. S. Geol. Survey. Bull. 479, 31 pp.—In order to obtain from a water analysis a full idea of the character of the water it is better to put less emphasis upon the concs. expressed as parts per mil. and more upon the percentages of the various equivalents present, both radicles and salts. The % equivs. are simplified by making the following groups: (1) Alkali equivs., (2) alk. earth equivs., (3) H equivs., (4) sulfate + chloride equivs., (5) carbonate + bicarbonate equivs. Any water may be classified in a broad way according to the relative proportions of the 5 groups as possessing salinity of the alkalies, or of the alk. earths, or alkalinity of the alkalies or of the alk. earths, or stated proportions of more than one of these qualities. A large number of analyses of waters from various sources are stated in these terms and their relation to the rocks through which they flow shown. The presence of the alk. carbonates is shown to be very favorable to the soln. of SiO<sub>2</sub> and the common assumption that humic acids act thus is discarded as out of harmony with the facts.

R. C. Wells.

Investigations on Water from Springs. J. J. Hofman. Gravenhage. *Pharm. Weekblad*, 48, 1003-18.—Careful analyses of chief mineral springs in Europe.

V. E. HENDERSON.

The Chemical Composition and Biological Content of Water. J. König, J. Kuhlmann and A. Thienemann. Landwirts. Versuchstation, Münster. Z. Nahr. Genussm., 22, 137-54.—An extended investigation of various types of surface waters to decide whether a biological exam. may suffice for judging its degree of purity and kind of pollution. Waters of low content of inorganic and organic material, of high inorganic material chiefly chlorides, of high organic, and of high pollution with both forms were investigated. The types of biologic life were detd. in many streams polluted in various ways. The larvae of the fly Ephydra riparia is an indication of salt H<sub>2</sub>O, and the Sphaerotilus, Beggiatoa and Tubificien of a water high in organic matter. It was found impossible to use this means without chem. data in judging the type of pollution.

D. S. Pratt.

Daytona Municipal Water Softening Plant. Geo. A. Main. Mun. J. Engr., 31, 463-5.—In a region of hard ground water containing H<sub>2</sub>S the intermittent system was adopted, using 3 reservoirs, as agitating and settling basins. The water is aerated, milk of lime added and 10 min. later soda ash soln. added. Compressed air is used for agitation. Each 1000 gals. of well water requires 2.4 lbs. lime and 0.67 lbs. soda ash. 75-90% saving in soap results. Algae growths have been prevented.

L. PEARSE.

The Operation of the Pittsburgh Water Purification Works. GEORGE A. JOHNSON. Eng. Record, 64, 447-8.—Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> and baffles in the basins are recommended to prepare the water. Filters may be started after cleaning, at the full rate if "hypo" be used. Oil wastes, probably paraffin, interfere with the proper operation of the filter.

LANGDON PEARSE.

Rapid Filter Plant at Minot, North Dakota. Eng. Record, 64, 408-9.—An engineering description of a filter plant of com. type. The coagulants used are: Fe (1.5 grains per gal.) and lime (5 grains per gal.) with a finish of "hypo" (0.2 grain per gal.).

LANGDON PRARSE.

Notes on Goulburn Water with some Experiments on its Clarification. G. J. Burrows. J. Proc. Roy. Soc., N. S. W., 43, 394-8.—Goulburn, New South Wales, is supplied with Wollondilly river water, which contains suspended matter so finely divided that storage and present filters are not always adequate. Lab. expts. showed that during one turbid condition 75 pts. per million com. Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> clarified the water.

Aluminium Sulfate. Eng. Record, 64, 458.—Specifications for purchase of Al<sub>2</sub>(SO<sub>4)</sub> as a coagulant at Bangor, Me., filter plant (cf. C. A., 5, 3487). LANGDON PEARSE.

The Determination of the Amount of Dissolved Oxygen Absorbed by Sewage Rffluents Containing Nitrites and of the Amount of Nitrites in Sewage Effluents and Water. R. W. CLARKE. Analyst, 36, 393-6.—The O-absorbing power was detd. by the following method: Dil. the effluent with 2-10 vols. of good tap water and shake thoroughly. Completely fill about 5 bottles of a known capacity (about 300 cc.) with the dil. sample. Det. the amt. of dissolved O present at the start and after 1, 2, and 5 days resp. by adding 1 cc. of 33% MnCl to the bottom of the bottle, followed by 3 cc. of 33% NaOH containing 10% KI. Shake, allow to stand for some time, atmospheric air being excluded. When the mixed hydrate of Mn has settled add 3 cc. of HCl and shake again. Neutralize with MgCO, and after the reaction is complete pour into a flask and titrate the free I with Na, S,O,. The diff. between dissolved O at the beginning and after a given interval is the O absorbed. The assumption that there is practically no change in the amt. of nitrites present has been justified. The method as applied to the estn. of nitrites is as follows: Fill a tall 500 cc. bottle to within 20 cc. of the top with a measured quantity of the sample. Add 4 cc. of 33% MnCl, and 12 cc. of the 33% NaOH containing 10% KI, shake thoroughly and let stand over night. Fill a 300 cc. bottle from the other bottle (avoiding aeration), add 3 cc. of HCl, neutralize with MgCO<sub>a</sub> and titrate back the liberated I with Na<sub>2</sub>S<sub>2</sub>O<sub>2</sub>. On known samples containing 0.04-0.6 pts. per 100,000 of N results within 5% of the amt. of N present were obtained.

C. P. WILSON.

Sewage Disposal and Sludge Production at Leeds. G. A. HART. Eng. Record, 64, 415.

LANGDON PEARSE.

Sewage Disposal. W. J. DIBDIN. Chem. Trade J., 48, 610-3.—Oxidation must be used sooner or later so why not start with it. Chem. pptn. and sludge drying are undesirable. Aerobic treatment is better, cheaper, does not cause a nuisance and the humus obtained has the highest manurial value consistent with economical and inoffensive treatment.

E. Bartow.

Chicago Sewage Disposal Experiment Station. Mun. J. Engr., 31, 421-6.—Details of the plant are given. The analyses of the crude sewage are given, with comparative results of other cities. Conclusions: The velocity of flow should not be less than  $\frac{1}{2}$  ft. per sec. in the grit chamber; a septic tank effluent has no advantage over a sedimentation effluent. Sludge digestion tanks are offensive. The biolytic tank generates  $H_2S$ . The Emscher tank gives a well clarified easily oxidized effluent, and a sludge more nearly approximating humus. Sprinkling filters can be used uncovered. Stone  $\frac{1}{4} = \frac{1}{4}$  in. size is most desirable, in depths of 6 ft. and over. A yield of  $\frac{2-2}{2}$  mil. gals. per acre can be obtained. No advantage is apparent in a fine-grained surface on a coarse body in a sprinkling filter.

Klur, H.: Untersuchung des Wassers an Ort und Stelle. 2 Aufl. Berlin: J. Springer. 150 pp., 4 M.

KOLKWITZ, R. AND SCHMIDTMANN, A.: Wasser u. Abwasser. Leipzig: 8°, 410 pp., 15 M.

RISTENPART, E.: Das Wasser in der Textil-Industrie. Leipzig: Max von Criegern. I M.

- U. S., 1,001,935, Aug. 29. O. A. EBERBACH, Ann Arbor, Mich. Water-softening compound, consisting of Na<sub>2</sub>PO<sub>4</sub> 75, (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> 8 and Na<sub>2</sub>CO<sub>2</sub> 17 parts.
- U. S., '1,001,964, Aug. 29. E. LEONHARDT, Bärwinkel, near Nauhardenberg, Ger. Assignor to Gebr. Heyl and Co. A. G., Charlottenburg, Ger. Preparing a disinfectant and deodorizer by distilling off a mixt. of cresol containing not more than 10% of phenol with a conc. soln. of Cu acetate in H<sub>2</sub>O, then treating the distillate with an acid Al sulfate and neutralizing the excess acid by adding Al<sub>2</sub>O<sub>2</sub>.Fe<sub>2</sub>O<sub>3</sub>.
- U. S., 1,002,547, Sept. 5. G. WICHMANN, Berlin, Ger. Assignor to Chem. Fabrik auf Actien (vorm. E. Schering), Berlin, Ger. Silver salt-ethylenediamine-albumose compounds, brownish powders readily sol. in H<sub>2</sub>O with an alk. reaction, giving no ppt. with reagents for Ag, carbonizing on heating and from which the organic base is driven off by distillation; made by causing albumose which is sol. in H<sub>2</sub>O and derived from meat albumen to act upon a double compd. of a Ag salt, e. g., AgNO<sub>3</sub>, and ethylene-diamine; used as antiseptics.
- U. S., 1,002,548, Sept. 5. Idem. Silver nitrate-ammonio-albumose, a brownish powder containing 7% Ag, dissolving easily in H<sub>2</sub>O with an alk. reaction and giving off NH<sub>2</sub> on distilling the aq. soln. with caustic alkali; made by causing albumose sol. in H<sub>2</sub>O but insol. in alc. to react upon silver nitrate-ammonia; similar products being also obtained from the NH<sub>2</sub> compds. of sulfate, phosphate, oxide, carbonate or chloride of Ag. The products may also be made by dissolving albumose in NH<sub>4</sub>OH and then adding the salt of Ag.
- U. S., 1,002, 603, Sept. 5. E. E. M. PAYNE, Leicester, England. Purifying water by adding to it a soln. of Na<sub>2</sub>PO<sub>4</sub> containing an excess of NaOH, the NaOH being sufficient to combine with the CO<sub>2</sub> and acid carbonates contained in the H<sub>2</sub>O, and the Na<sub>2</sub>PO<sub>4</sub> forming normal phosphates of the metals in soln. in the water and pptg. them.
- U. S., 1,003,191, Sept. 12 O. B. MAY, New York, N. Y. Assignor to Morgenstern and Co., New York, N. Y. Bismuth salt of iodized resorcinolsulfonic acid, having the approx. formula (C<sub>6</sub>H<sub>2</sub>(OH), ISO<sub>6</sub>)<sub>2</sub>Bi.3Bi(OH)<sub>2</sub>, an antiseptic, amorphous brownish powder, slightly hygroscopic, partly decomposable by H<sub>2</sub>O with the formation of Bi(OH)<sub>2</sub> and iodoresorcinolsulfonic acid, readily sol. with decomp. in conc. HCl, and upon being heated losing at first I, then charring and finally leaving a yellow residue of Bi oxide; made by reacting upon a sol. salt of iodized resorcinolsulfonic acid, e. g., the K salt, with a Bi salt, e. g., Bi(NO<sub>2</sub>)<sub>2</sub>, and neutralizing with NaHCO<sub>2</sub>.

Brit., 11,938, May 13, 1910. C. NEFF, 19 Georgstrasse, Hanover, Ger. Treating liquids and imparting detergent properties to water by causing it to travel in frictional contact with an extensive and active surface of Al scoured with sand, emery, or the like, and cleaned with caustic alkali or the like.

### 15. SOILS AND FERTILIZERS.

#### F. P. VEITCH.

The Law of the Minimum and the Inference Drawn from it. E. A. MITSCHERLICH. Landw. Vers. Stat., 75, 230-63.—The law of the minimum is not a simple but a logarithmic function. The plant expts. which have been a common measure for the value of fertilizers cannot therefore be used for this valuation. Expts. modeled after Hellriegel's sand cultures are advized. By the use of such methods, in which neither the

sand nor the fertilizer salts used as a basis can influence the nutrient existing as a minimum, comparative values can be derived for different fertilizers containing the same nutrients and for different plant cultures. These values depend moreover on the place and the season in which the expts. are conducted.

E. C. LATHEOP.

The Development of Marsh Soils. A. R. Whitson and F. J. Sievers. Univ. of Wis. Agr. Expt. Str., Bull. 205.
S. C. Clark.

The Gumbago Soils of Iowa. W. H. STEVENSON AND J. F. BARKER. Agronomy. Sec., Ia. State Coll. of A. & M. A. Expt. Sta., Bull. 119.—Analyses show in surface ft. of this soil 0.250-0.317% N, 2.25-3.63% C, 4.09-6.60% org. matter, 0.043-0.124% total P, and 0.66-8.84% CaO; and in one sample about 2 ft. below surface the figures given are as follows: 0.168%, 2.03%, 3.69%, 0.071%, and 1.60%, resp. S. C. C.

The Improvement of Sandy Soils. A. R. Whitson and F. J. Sievers. Univ. of Wis. Agr. Expt. Sta., Bull. 204.—The fertility needs of sandy soils are N, P, and K, supplied directly in com. fertilizers or indirectly in manure or other vegetable matter. Legumes and lime are also very important.

S. C. Clark.

The Management of Heavy Clay Soils. A. R. WHITSON AND E. J. DELWICHE. Univ. of Wis. Agr. Expt. Sta., Bull. 202.—Manure and other vegetable matter should be added to these soils together with P. Drainage and crop rotations are important. S. C. CLARK.

Plat Experiments with Fertilizers. J. H. STEWART AND H. ATWOOD. W. Va. Univ. Agr. Expt. Sta., Bull. 131.—Continuous tests for 11 yrs. show that the first limiting element is P, then N, and finally K, for rye, wheat, clover, corn, cowpeas, and timothy; but P, K, and N is the order for potatoes. Stable manure has been as effective as a complete fertilizer, especially in the later yrs. The use of lime has not been fully satisfactory, although in the later yrs. there seems to have been some benefit. "If leguminous crops are raised and either plowed under or fed on the farm and the resulting manure carefully saved and applied to the soil it is very probable that in practice it will be necessary to purchase only P in order to increase the productiveness of soils of this type and to maintain them in a condition of high fertility." S. C. C.

Analysis of Lime-nitrogen. H. Kappen. Chem. Ztg., 35, 950-2.—K. obtained the same results for total N in lime-N by the following modifications of the Kjeldahl method. (a) 30 cc. conc.  $H_2SO_4$ ; (b) 30 cc.  $H_2SO_4$  + 10 cc.  $H_2O_3$ ; (c) 30 cc.  $H_2SO_4$  + 1 g. salicylic acid + 5 g.  $Na_2S_2O_3$ ; (d) 30 cc.  $H_2SO_4$  + 2 g. salicylic acid + 2 g. Zn dust. He therefore concludes that contrary to the statements of Monnier (C. A., 5, 2793) no special modifications of the Kjeldahl method are necessary for such material. He was unable to confirm the results of Monnier regarding the effect of  $H_2O_3$  on lime-N. Cyanamide N was detd. by pptn. with 0.01 N AgNO3 (ammoniacal) and titrating the excess, with satisfactory results.

Sulfur as a New Fertilizing Material. MAIZERES. Engrais, 26, 685.—S in additions of 250-500 kg. per hectare has been found to give large increases in the yield of potatoes and beets. Its action has been attributed to its disinfecting action, but it also enters into the comp. of the plant.

E. C. LATHROP.

The Action of Potash on the Orgnaic Nitrogen of Humid Soils. R. POINTU. Rev. agric.; through Engrais, 26, 691.—In peat soils lime is used to correct the acidity so that nitrification will take place. But often peat soils are high in lime and still have no nitrification. If potash is added nitrification takes place readily. The analysis of such soils often shows that they are poor in potash, but rich in non-nitric N. Soils rich in potash are generally poor in organic N. Lime is indispensable for the changing of organic N to NH<sub>2</sub> nitrogen and potash is essential for the formation of nitric N.

E. C. LATHROP.

\*Commercial Fertilizers. BLTON FULMER. State Coll. of Wash. Expt. Sta., Bull. 98.

S. C. Clark.

Analyses of Commercial Fertilizers. M. B. HARDIN, et al. Clemson Agr. Coll. S. C. Expt. Sta., Bull. 154.—Analyses of 1188 official and 98 other samples.

S. C. C.

Inspection of Commercial Fertilizers. P. F. TROWBRIDGE. Univ. of Mo. Agr. Expt. Sta., Bull. 85. S. C. C.

Inspection and Analyses of Commercial Fertilizers. W. F. Hand, et al. Miss. Fertilizer and Feeding Stuff Control Agr. Expt. Sta., Circ. 32.—The first of 3 reports covering yr. 1910-11.

S. C. C.

Analyses of Commercial Fertilizers, 1911. F. W. Woll. Univ. of Wisc. Agr. Expt. Sta., Circ. 25. S. C. C.

Organic Phosphoric Acid of the Soil. G. S. FRAPS. Texas Agr. Expt. Sta., Bull. 136.—This is a report of expts. and analyses, tending to controvert Stewart (C. A., 4, 2702) and leading to the conclusion: "We have at present no method for est. the organic P of the soil."

S. C. C.

Commercial Fertilizers. B. H. HITE AND F. B. KUNST. W. Va. Univ. Agr. Expt. Sta., Bull. 132.—A complete report for 1910.

S. C. C.

The Adulteration of Anhydrous Ferrous Sulfate Used in Agriculture. A. Gioux and A. Dupuy. Ann. fals., 4, 443.—The use of anhydrous FeSO<sub>4</sub> for the destruction of charlock failed this year because of its adulteration with FeSO<sub>4</sub>.7H<sub>2</sub>O, powdered CaSO<sub>4</sub> wood ash, and white sand. To insure efficiency, the water content should not exceed 10% and the insol. matter 2%.

D. S. Pratt.

Peach Leaf Curl. E. WALLACE AND H. H. WETZEL. Cornell Univ. Agr. Expt. Sta., Bull. 276.—Comp. of special sprays, for controlling this disease. S. C. CLARE.

Spraying for Black Rot of the Grape in a Dry Season. D. REDDICK, C. S. WILSON, AND C. T. GREGORY. Cornell Univ. Agr. Expt. Sta., Bull. 296. S. C. CLARK.

Woody Aster. O. L. PRIEN AND L. CHARLES RAIFORD. Univ. Wyoming Agr. Expt. Sta., Prelim. Bull. 88.—The woody aster, Xylorrhiza parryi Gray, grows only on alkalied, gumbo-clay soils and is poisonous to sheep. Clinical data is given.

S. C. CLARK.

The Bradication of Wild Radish by Sulfuric Acid. E. RABATÉ. J. agric. prat; through Engrais, 26, 607.—The action of a 1% soln. of  $H_2SO_4$  is much more rapid than either that of FeSO<sub>4</sub> or CuSO<sub>4</sub>. The acid also attacks the minerals of the soil and the humus. 10 hectoliters per hectare of 1% soln. is advized. This soln. will kill the weeds in  $\frac{1}{4}$  hr. and insects are killed at once; wheat is affected but fully recovers in 10 days.

E. C. LATHROP.

Citrus Fruit Insects. H. J. QUAYLE. Univ. of Calif. Agr. Expt. Sta., Bull. 214.

—Fumigation with HCN is recmmended, and the details of the procedure are given.

NaCN may displace KCN on account of cost. Formulas for both are given. NaCl as an impurity gives rise to HCl which is detrimental.

S. C. CLARK.

Orchard and Garden Spraying. A. G. RUGGLES AND E. C. STAKMAN. Univ. of Minn. Agr. Expt. Sta., Bull. 121.—Formulas for 25 sprays. S. C. C.

Orchard Sprays and Spraying. A. B. CORDLEY AND H. S. JACKSON Oreg. Agr. Coll. Expt. Sta., Bull. 13.—Formulas. S. C. C.

Spraying Cucumbers and Cantaloupes. T. C. Johnson. Va. Truck Expt. Sta., Bull. 5.—Formulas of special sprays. S. C. C.

Valuation of Calcium Nitrite and Nitrate (STUTZER, GOY). 7.

Preparation of Neutral Ammonium Citrate Solutions (HALL). 7.

Ammonia and Nitric Acid in Rain Water (AUFRAY). 14.

Manufacture of Nitrogen Compounds (Scorr). 4.

Nitrogen in Commercial Ammoniates (RUDNICK, et al.). 7.

Alkalinity of MgNH<sub>4</sub>PO<sub>4</sub> and Acidimetry of NH<sub>4</sub> Phosphomolybdate (HUNDES-HAGEN). 7.

Alkali in Arsenical Dip-fluids (COHEN).7.

CAMERON, H. E.: The Soil Solution: the Nutrient Medium for Plant Growth. Easton, Pa.: Chemical Pub. Co.: 8°, 136 pp., \$1.25.

KÖNIG: Die Untersuchung landwirtschaftlich und gewerblichwichtiger Stoffe. 4 Aufl. Berlin: P. Parey. 35 M.

RUSSELL, E. J.: Lessons on Soil. London: 12°.

- U. S., 1,001,350, Aug. 22. N. CARO and T. E. SCHERLE. Making a stable, non-hygroscopic ammonium phosphate fertilizer; see C. A., 5, 358.
- U. S., 1,001,852, Aug. 29. R. H. HUTCHINSON, New York, N. Y. Insecticide and fungicide, consisting of saponified wool grease, an As compd. and the mixt. produced by b. Ca(OH), and S together in H<sub>2</sub>O and concg. the resulting liquor.
- U. S., 1,002,143, Aug. 29. F. W. FRERICHS, St. Louis, Mo. Making calcium metaphosphate and ammonia by roasting a mixt. of 1 mol. proportion of Ca phosphate and 2 mol. proportions of  $(NH_d)_8SO_4$ , gradually raising the temp. from normal to above 343° until the major portion of the  $NH_2$  has been liberated and the major portion of the Ca phosphate has been converted into Ca metaphosphate.
- U. S., 1,002,182, Aug. 29. H. D. RUHM, Mountpleasant, Tenn. Apparatus for washing and recovering fines from phosphate rock.
- U. S., 1,002,198, Aug. 29. F. W. Frerichs, St. Louis, Mo. Making calcium pyrophosphate and ammonia by roasting a mixt. of Ca phosphate 1 and  $(NH_4)_2SO_4$  1 mol. proportion, gradually raising the temp. from normal to above 343° until the major portion of the NH<sub>2</sub> has been liberated and the major portion of the Ca phosphate has been converted into Ca pyrophosphate.
- U. S., 1,002,247, Sept. 5. C. ELLIS, Montclair, N. J. Agricultural spray for use as an insecticide and fungicide, formed from a partially oxidized soln. of a Ca polysulfide, As sulfide, KOH or K<sub>2</sub>S and preferably also waste sulfite liquor. Sulfides of Ba, K or Na may be used instead of Ca polysulfide.
- U. S., 1,002,248, Sept. 5. *Idem*. Making a fertilizer by mixing humus material with basic slag, substantially sterilizing the humus material by the addition of borax waste and inoculating the material with N-assimilating bacteria.
- U. S., 1,003,273, Sept. 12. W. R. KLECKNER, Bay City, Mich. Fertilizing scale-poison for injection into the trunks of trees, formed of KCN 95, FeCl<sub>8</sub> 45, Fe(NO<sub>2</sub>)<sub>8</sub> 50, H<sub>2</sub>PO<sub>4</sub> 5 and bread crumbs 5 parts.
- U. S., 1,002,297, Sept. 5. C. N. MERIWETHER, Trenton, Ky. Rendering the phosphoric acid in phosphate rock available by pulverizing the rock and mixing it with a mixt. of Fe oxide ore and Fe and then heating to a temp. below fusing and thoroughly agitating during the heating. The mixt. is maintained at a red heat for 1-6 hrs.
- U. S., 1,003,541, Sept. 19. W. F. K. STOCK, Darlington, England. Fertilizer, insecticide and fungicide, consisting of spent oxide of Fe, containing free S, from gas

works, I, C waste from alkaline liquors obtained in paper mfg. I and crude drained  $C_{10}H_{8}$  I part.

U. S., 1,003,681, Sept. 19. R. WILLIAMS, Birmingham, Ala. Making superphosphate by treating ground phosphate rock with a quantity of H<sub>2</sub>SO<sub>4</sub>, which has been artificially chilled (preferably to 0-37°), sufficient only to convert the Ca<sub>4</sub>(PO<sub>2</sub>)<sub>2</sub> into sol. phosphate and artificially lowering the temp. of the reacting mixt. to about 37° or lower to avoid reaction of the acid on impurities in the rock. The rock also may be chilled if desired before mixing with the acid.

Ger., 236,491, Aug. 2, 1910 Addition to 224,077, Dec. 7, 1909; cf. C. A., 4, 3287. A. HAUCK, Saarbrücken. In obtaining citric acid soluble phosphoric acid from crude phosphates, the phosphates, heated with CO<sub>3</sub>, are further heated with access of air instead of under CO<sub>2</sub> pressure, or they are fused.

Ger., 236,850, July 3, 1910. G. CUBATELLI, Toranto, Italy. Superphosphate; see C. A., 5, 2889.

## 16. FERMENTED AND DISTILLED LIQUORS.

#### ROBERT WAHL.

The Alcoholic Content of Spirits. K. v. Buchka. Berlin. Z. Nahr. Genussm., 22, 34-43.—The question of the relation between alc. content of distilled spirits and physiological action is very complex. Because of increased taxation, a tendency is observed to reduce the alc. and cover the resulting mild taste artificially. MeOH is also used. Analyses of various brands from different localities are given, showing considerable variation in alc.

D. S. Pratt.

Balling Formula for the Calculation of Original Gravity. F. SCHOENFELD. Wockschr. Brau., 28, 209-12, 221-4.—The author has experimented with the Balling formula and redetermined the factor for bottom fermentation. The formula now reads p = (2.0195A + n)/(1.0195A + 100) 100, in which p = 000 original density, A = 010 and n = 0211 remaining extract %. The results obtained by the well-known B. formula in case of top fermentation tend to be low, which may be due to loss of alc. by evapn. and greater yeast production.

C. A. Nowak.

The Influence on the Extent of Multiplication of Variation in the Amount of Yeast Added to the Wort. F. HAYDUCK AND G. ANDERS. Wochschr. Brau., 28, 233-6.— The authors conclude from their expts. that the absence of growth when large amts. of yeast are seeded, is not to be attributed to the accumulation of the alc. formed nor to the disappearance of sugar. They find that the single cells increase in size without budding owing to the fact that there is a lack of space necessary for multiplication.

C. A. NOWAK.

The Detrimental Effect of Moisture Content on the Value of Color Malt. G. Jacob. Wochschr. Brau., 28, 341-4.—The author has conducted expts. to det. the effect of moisture content upon the coloring power and purity of flavor of color malt. Calculations show that for every 1% of additional moisture 1.11% more color malt will be required to produce the same color in the product. The influence of increased moisture content upon the yield of extract is inappreciable. This holds equally true of the reduction in value by reduced % of extract. At the same time it must not be concluded that the detn. of moisture content and % of extract is an unimportant matter, for reason which the author proposes to give later.

C. A. Nowak.

Top Fermenting Yeast and Acidity. P. PETIT. Brasseri et malterie; through Wockschr. Brau., 28, 395-6.—The treatment of yeast with phosphoric acid has nothing

in common with the customary washing of yeast either with tartaric acid or various antiseptics. The phosphoric acid acts upon the functions of the yeast, imparting to it a specific power and at the same time rendering it immune to disease. It is advizable to det, the proper amt, required in each brewery, thereby providing a simple means for regulating the acidity of worts and compensating for any lack of acid substances in the raw materials and in the deficient comp. of the brew water. The following method of treatment is recommended: To the yeast  $1-2^{1}/2$  times its weight of water is added containing about 1.4-2.5 g. acid per l. depending upon the content of carbonates in the water. After about 30-40 min, the whole can be used for pitching. Aside from other advantages derived from the acidity in the manuf. of beer, the acidity of the wort owing to the N comp, dissolved therein, regulates the yield of yeast, and renders the beer more chillproof and more durable.

C. A. Nowak.

Ozone in the Brewery. L. VETTER AND E. MOUFANG. Wockschr. Brow., 28, 377-82.—Proper ventilation in the cellars necessitates a large volume of air, good draught and low conc. of O<sub>3</sub> (approx. 0.0003-0.0008 g. per cm.). For the refrigerator room with open wort cooler on the other hand a high conc. is required, the maximum output of 0.001 g. of O<sub>3</sub> per cm. of air being necessary to maintain the wort in a steril condition at incubation temp. Furthermore ozone is not to be regarded as a substitute for fresh air, but as a supplementary factor in fresh air ventilation. Sterilization in contrast to ventilation requires a high conc. of O<sub>3</sub> and a small vol. The author further describes methods of application of O<sub>3</sub> to the various cleaning and sterilizing purposes in the brewery.

C. A. Nowak.

Determining Nitrogen in Liquid. O. FÜRNROHR. Wochschr. Brau., 28, 257-8

—In detg. the N content of a wort considerable trouble is experienced in boiling down owing to frothing and danger of carmelization. The author condemns the practice of adding yeast prior to conc. since yeast contains N. He recommends conc. at uniform temp. of 150°; by this method wort can be evapd. to a syrup without any frothing or loss of N by carmelization. Uniform results are thus obtained, which is not possible if wort is evapd. over naked flame at 200°.

C. A. Nowak.

Chemical Behavior and Preservative Action of Sulfites in Beer. J. L. Baker AND F. E. Day. J. Inst. Brewing, 17, 465-77.—Inasmuch as the sulfites affect the flavor of beer they should, if employed, be added in as small quantities as possible. The development of acidity is not furthered by large quantities of sulfites. For purposes of preservation an addition of S corresponding to 50 pts. of SO<sub>2</sub> per mil., and for favoring the development of acidity 100-200 pts. SO<sub>2</sub> per mil. give the max. beneficial effect. When beer is treated with sulfite the same appears to combine slowly with some of the constituents of the beer and inasmuch as these compds. (sulfonates or sulfurous esters) appear stable and inactive it is likely that they have no antiseptic value. The antiseptic value of the sulfites is probably due to the part remaining uncombined or to those compds. which are hydrolyzed by the action of water. Even small quantities of sulfites have a retarding effect on the secondary fermentation.

C. A. NOWAK.

On the Use of Aluminium in the Construction of Brewery Vessels. O. W. WILLCOX, L. KUNZ AND R. JUERST. Pure Products, 7, 445-50.—Data on the solvent action of beer, ale, etc., on Al and of the effects of various cleansing and disinfecting solns. on Al are given. The solvent action of beer and ale on Al under lab. conditions is also stated.

W. D. Biorlow.

The Hearing on Beer before the Board of Food and Drug Inspection. Pure Products, 7, 455–65. W. D. B.

The Judging of Distilled Spirits. A. JUCKENACK. Berlin. Z. Nahr. Genussm.,

22, 47-54.—Rules governing the content of alc. and the methods for manufg. cognac, rum, arrak, fruit brandies, bitters, liqueurs, corn brandy, etc., are given.

D. S. PRATT.

Behavior of Yeast Enzymes (EULER, KULLBERG). 11.

Precision Fermentation Saccharometer. 1.

Enzymes of Diastase (LYALIN). 11.

Effect of Alcohol Denatured with Wood Spirit on Health (HORBACZEWSKI). 22.

HEIDE, C. AND JAKOB, F.: Praktische, Uebungen in der Weinchemie und Kellerwirtschaft. Stuttgart: E. Ulmer. 8°, 121 pp., 2.60 M.

WEINWURM, E.: Betriebestörungen in der Malzfabrikation und Bierbrauerei. Hannover: M. Jänecke. 3 M.

# 17. PHARMACEUTICAL CHEMISTRY.

#### VIRGIL COBLENTZ.

Assay of Cinchona Bark—Estimation of Quinine and Total Alkaloids. VIGNERON. J. pharm. chim., 3, 103-8.—An abbreviated form of the codex assay method is recommended for proximate results. Ext. with 0.01% HCl, concentrate so that 100 cc. represents 20 g. of bark and ppt. an aliquot with a weak soln. of NH<sub>4</sub>OH and NaOH previously satd. with the alkaloids from the assay. Wash the ppt. sparingly, dry below 45°, dissolve in Et<sub>2</sub>O-CHCl<sub>2</sub> and then evap., dry and weigh the total alkaloids. To det. quinine, dissolve it out of the total alkaloids, acidify with dil. H<sub>2</sub>SO<sub>4</sub> ppt., dry, and weigh it as the chromate.

V. K. Chesnut.

Identification of Diethylbarbituric Acid (Veronal). A. JORISSEN. J. pharm. chim., 3, 478-81; Ann. chim. anal., 16, 370-3.—Fuse about 3 g. KOH in a Ni crucible over a small flame. When melted, add in small quantities 0.3 g. veronal and continue heating 2 mins. Dissolve the mass when cold with 10 cc. H<sub>2</sub>O. Half of this should give the Prussian blue cyanide test with FeSO<sub>4</sub>. Acidify the remainder with H<sub>2</sub>SO<sub>4</sub> and note escape of CO<sub>2</sub>. Shake well with Et<sub>2</sub>O and evap. the ext. Oily drops with an odor like rancid butter are left. Tested with dil. FeCl<sub>2</sub> it should give the thiocyanate test. When veronal is mixed with 5 parts CaO and lighted on Pt foil the vapor burns and the residue becomes red and it will remain red if the flame be suddenly extinguished.

V. K. C.

Kolatein, a Second Phenolic Constituent of Cola. A. Goris. Bull. sci. pharmacolog., 18, 138-40; Pharm. J., 86, 809.—Under the name kolatein, G. describes a new cryst. compd. similar to phloroglucin and kolatin. The larger crystals of kolatein (1 cm. × 2-5 mm.) may be sepd. by hand from the kolatin after crystallization from water. The compd. is free from ash, does not liberate CO<sub>2</sub> from KHCO<sub>3</sub>, is pptd. by Pb(C<sub>2</sub>H<sub>2</sub>O<sub>2</sub>)<sub>2</sub>, colored green by FeCl<sub>3</sub> and the soln. becomes violet red with NH<sub>4</sub>OH. It is better and it gives a green color with FeCl<sub>3</sub>. M. 257-8°. The exact nature of the compd. has not yet been detd.

V. K. C.

Composition of Gentian Tinctures Prepared from Unfermented Gentian Roots with Hot and Cold Alcohol. M. BRIDEL. J. pharm. chim., 3, 534-9.—With Bourquelot, B. previously showed that when gentian root is dried in the shade, all of the gentiopicrin is conserved and that the entire lack of this glucoside in the com. root is due to the fermentation to which it is subjected before drying. B. now finds that tinctures of gentian made with 60% EtOH from unfermented roots are also devoid of gentio-

picrin while tinctures made by throwing the powdered root into b. 60% EtOH contain practically all of the gentiopicrin found in the unfermented root. The difference between the 2 tinctures is most strikingly shown in the diff. in the optical deviation (—43' in the one made with cold EtOH and —4°7' in the other). Expts. are cited which show that this loss of glucoside is due to the hydrolytic action of the emulsin of the root upon the gentiopicrin. 60% EtOH retards the action but little, this being also shown to be the case with invertase and sucrose. Gentiogenin, the hydrolysis product of the action of emulsin on gentiopicrin, being insol. in H<sub>2</sub>O crystallizes out from tinctures made with cold EtOH and may be detected thus: Dissolve the crystals in 3-4 cc. of 95% EtOH in a test tube, add drop by drop, to prevent mixing, an equal vol. of H<sub>2</sub>SO<sub>4</sub> and note the blue zone at the point of contact. When treated with emulsin the hot EtOH ext. easily gives this test, while with the cold EtOH ext. little or no blue color is obtainable. Gentian tinctures prepared according to the Fr. Codex contain no gentiopicrin.

Contribution to the Study of the Adsorption Products of Iodine. LAMBERTO CORRIDI. Ist. Stud. Sup. Firenze. Arch. farm. sper., 12, 265-75.—From a mixt. containing 3 g. animal charcoal, 3 g. I and 30 cc. solvent (EtOH, Et<sub>2</sub>O, CHCl<sub>2</sub>, CS<sub>3</sub>, and a mixt. of the first three), the charcoal adsorbs in 24 hrs. 4.87-7.50% I. The adsorption product is stable at 120° and gives no color with starch paper. I is gradually liberated, however, on treatment with organic solvents and dil. acid or alkali. A. W. Dox.

Superiority of Fermi vaccin to Pasteur vaccin. F. M. MARRAS. Univ. Sassari. Arch. farm. sper., 12, 298-316. A. W. Dox.

Simple Determination of Ethyl Nitrite in Spirit of Nitrous Ether. O. Herting. Pharm. Zig., 56, 423; through J. Soc. Chem. Ind., 30, 922.—To 10 cc. H<sub>2</sub>O and 5 cc. KClO<sub>3</sub> satd. soln. in a stoppered flask are added 5 cc. of the sample followed by 5 cc. HNO<sub>3</sub> (10%) and the mixt. shaken frequently for 30 mins.; 10 cc. o.1 N AgNO<sub>3</sub> are added and the excess titrated with o.1 N NH<sub>4</sub>SCN. Each cc. o.1 N AgNO<sub>3</sub> represents 0.0255 g. C<sub>2</sub>H<sub>4</sub>NO<sub>3</sub>.

H. C. FULLER.

Experiments with the Cat Method for Testing Digitalis and its Allies. C. R. ECKLER. Am. J. Pharm., 83, 478-91.—The method is complicated, requires more time than other methods and is impracticable. The results are variable, sometimes checking closely, again not. Lactating animals cannot be depended upon as they seem to possess a greater tolerance for the drug, the degree depending on the stage of lactation.

H. C. F.

The Ash Content of Drugs. M. I. WILBERT. Am. J. Pharm., 83, 474-8.—The detn. of the ash content of official drugs is important and practicable with gums, resins, pollen grains, seeds, spices and powdered drugs. It is not generally applicable to leaf drugs, roots and barks because of the difficulty of sampling. Pharmacopeial requirements respecting ash content should be based on the air-dried drug as this is the com. form and that generally used in making galenical preps., and in order to secure uniform results there must be a minutely described method.

H. C. F.

A Note on the Assay of Formaldehyde. E. Elvove. Am. J. Pharm., 83, 455-71.—The minimum requirement of the U. S. P. should be 36% instead of 37%. The KCN method might with advantage be substituted for the  $H_2O_2$  method and is applicable to the U. S. P. soln. by weighing 0.5 cc. into a 150 cc. Erlenmeyer, introducing 100 cc. 0.1 N KCN, mixing with 40 cc. 0.1 N AgNO<sub>2</sub> and 10 cc. dil. HNO<sub>2</sub> in a 200 cc. graduated flask, filtering 100 cc. and titrating the excess Ag with 0.1 N KSCN.

H. C. F.

Tincture of Cantharides. E. G. EBERHARDT. Am. J. Pharm., 83, 471-4.— Cantharides can be exhausted by gently heating with NaOH, drying, and extracting with dil. EtOH, but the resulting prep. is weak in vesicating properties. An active produce may be obtained by liberating with acid and extracting with Me<sub>2</sub>CO.

H. C. F.

Detection of Impurities in Amidopyrine [Pyramidone, Dimethylaminoantipyrine]. C. Kollo. Pharm. Post, 1911, 173; through J. Soc. Chem. Ind., 30, 766-7.—Alkali halides and unchanged antipyrine may be present. The former cannot be detected by the ordinary AgNO<sub>3</sub> test as the substance itself gives an opalescence. Günzburg's reagent, consisting of one part each of phloroglucinol and vanillin in 500 parts Et<sub>2</sub>O, will show very small quantities. The sample is ignited on a Pt wire and the products of combustion brought in contact with the residue from 15-20 drops of the reagent, when a red ring or area will appear in the presence of halides. Another test consists in mixing 0.1 g. with 0.2 g. each of AgNO<sub>3</sub> and Cu(NO<sub>3</sub>)<sub>3</sub>, heating to redness in a tube closed at one end, treating with dil. H<sub>2</sub>SO<sub>4</sub> and Zn, filtering and adding AgNO<sub>3</sub> and HNO<sub>3</sub> which will give a ppt. if a halide is present. Antipyrine is detected by adding 0.1 g. to 10 drops of a soln. of 1 g. p-dimethylaminobenzaldehyde in 5 cc. HCl and 94 cc. EtOH, evapg. and noting the appearance of a firey red ring.

Resence of Bupleurum fruticosum Linn. L. Francesconi and G. Sanna. Ist. chim. gen. r. univ. Cagliari. Gazz. chim. ital., 41, 1, 798-813; cf. C. A., 5, 3467.—From a careful fractionation of the essence it is found to contain a large amt. of a terpene which is a mobil, colorless, refractive liquid of pleasant odor and pungent, bitter taste,  $d_{14}$  0.8416,  $[\alpha]_1$  35.7°, b. 167-9°, n 1.4862, polymerizes at 200°, forming a white, amorphous solid, m. 90-100°, becoming yellow,  $[\alpha]$  —66.14°. There is also present in the essence an alc. and an ester (apparently new). With Br and HCl, the terpene yields viscous products; with NOCl a minute amt. of a cryst. nitrosochloride, begins to m. 97°, decomp. 102°, and a dextro-rotatory compound, lighter than  $H_2O$ , b. about 200°, is oxidized by AgNO<sub>4</sub> more easily than carvone, and gives a semicarbazone, silvery scales, m. 197-8°, d-rotatory and very stable. These facts indicate that the terpene is a compd.  $C_{10}H_{16}$  of the limonene series.

Action of Nitrosyl Chloride on the Essence of Bupleurum fruticosum. Nitrosochlorides. L. Francesconi and E. Sernagiotto. Ist. chim. r. univ. Cagliari. Atti accad. Lincei, 20, II, 190-6.—After repeated failures to obtain a nitrosochloride from the essence by Wallach's method (Ann., 253, 110), it was found that 0.4-1.25 g. nitrosochloride, depending on the fraction of essence used, could be obtained from 5 cc. of the latter by discarding the use of  $H_2O$  and AcOH and carrying out the reaction in alc. HCl. The nitrosochloride m. 92-4°, is faintly blue when fresh but rapidly decomp., forming a reddish product of unpleasant odor,  $[\alpha]$  about  $-205^{\circ}$  whether prepared from fractions of essence showing  $[\alpha]$  53° or 19°. By fractional crystn. from CHCl<sub>2</sub> it can be sepd. into an  $\alpha$ -form, m. 101-2°,  $[\alpha]_D$   $-175^{\circ}$ , very sol. in CHCl<sub>3</sub>, and a  $\beta$ -form, m. 100-1°,  $[\alpha]_D$   $-285^{\circ}$ .

Codeine Oxide (Freund, Speyer). 10. Strychnine and Brucine (Ciuba, Scagliarini). 10. Reaction for Formic Acid (Deniges). 7.

BIECHELE, M.: Anleitung zur Erkennung und Prüfung aller im deutschen Arzneibuch. 13 Aufl. Berlin: 8°, 615 pp., 6.60 M.

HOLFERT, J.: Volkstümliche Namen der Arzneimittel, Drogen und Chemikalien. 6 Aufl. Berlin: 8°, 266 pp., 4.60 M.

PARRY, E. J.: The Chemistry of Essential Oils and Artificial Perfumes. New York: Van Nostrand. 8°, 554 pp., \$5.00.

### RAMBOUSLE, J.: Gewerb iche Vergiftungen. Leipzig: Veit & Co. 12 M.

- U. S., 1,001,247, Aug. 22 H. C. BUER. Extracting lecithin from seeds of lupines and other pulses; see C. A., 5, 974.
- U. S., 1,001,829, Aug. 29. M. ENGELMANN, Elberfeld, Ger. Assignor to Farben. vorm. F. Bayer & Co., same place. Mercury compound probably having the formula

obtainable by reacting with alkali and diethylbarbituric acid upon o-oxymercuric salicylic anhydride, the product being a whitish cryst. powder, easily sol. in H<sub>2</sub>O to a neutral soln., insol. in alc., ether, C<sub>2</sub>H<sub>4</sub>, acetone and CHCl<sub>2</sub> and containing the Hg so firmly combined that the H<sub>2</sub>O soln. does not give any ppt. on the addition of dil. NaOH or cold (NH<sub>2</sub>)<sub>2</sub>S soln.; used hypodermically as an antisyphilitic.

- U. S., 1,001,933, Aug. 29. M. DOHRN and A. THIELE, Berlin, Ger. Assignors to Chem. Fabr. auf Aktien (vorm. E. Schering), Berlin. Methoxymethylsantalol, C<sub>10</sub>H<sub>20</sub>-OCH<sub>2</sub>, OCH<sub>2</sub>, a colorless liquid, b. 152-1584°, insol. in H<sub>2</sub>O, sol. in organic solvents, liberating CH<sub>2</sub>O on heating with mineral acids; used as a medicin; made by the reaction of chloromethyl ether upon santalol, in the presence of a condensing agent, or upon an alkali salt of santalol.
- U. S., 1,002,243, Sept. 5. P. EHRLICH and A. BERTHEIM, Frankfort a/M, Ger. Assignors to Farbwerke vorm. M. L. & B., Höchst a/M, Ger. Aminophenoxyarsenoxide, a whitish cryst. powder, scarcely sol. in H<sub>2</sub>O, readily sol. in dil. mineral acids, NaOH soln. and MeOH, scarcely sol. in ether and insol. in C<sub>4</sub>H<sub>4</sub>, yielding when treated with H<sub>2</sub>O<sub>2</sub>, aminophenoxyarsenic acid; made by treating amino-oxyarsenic acid with a weak reducing agent, e. g., SO<sub>2</sub>.
- U. S., 1,002,399, Sept. 5. F. HOFMANN and K. MEISENBURG, Elberfeld, Ger. Assignors to Farben. vorm. F. Bayer & Co., same place. Making  $\beta$ - $\gamma$ -dimethylerythrene by first distilling pinacone with the acid K salt of 1,5-naphthalenedisulfonic acid and then sepg. the  $\beta$ - $\gamma$ -dimethylerythrene.
- U. S., 1,002,400, Sept. 5. *Idem.* Making  $\beta$ - $\gamma$ -dimethylerythrene by first distilling pinacone with KHSO<sub>4</sub> and then sepg. the  $\beta$ - $\gamma$ -dimethylerythrene from the distillate and purifying it by redistillation over Na, NaHSO<sub>4</sub> or Na or K pyrosulfate may be used instead of KHSO<sub>4</sub>.
- U. S., 1,002,854, Sept. 12. O. LIEBKNECHT, Frankfort a/M, Ger. Assignor to Roessler & Hasslacher Chem. Co., New York, N. Y. Hydrogen peroxide solution containing 0.1% salicylic acid, phthalic acid or benzoic acid to render it stable.
- U. S., 1,002,909, Sept. 12. F. FLAECHER, Höchst a/M, Ger. Assignor to H. A. Metz & Co., New York, N. Y. Crystallized hydrochloride of o-dioxyphenylethanol-methylamine, made by treating the optically inactive base with alcoholic HCl and allowing it to crystallize, in the form of rhombic prisms, which are stable in air, color-less, readily sol. in H<sub>2</sub>O with neutral reaction, difficultly sol. in abs. alc., m. at 161°, the aq. soln. of the salt being optically inactive.
- U. S., 1,003,289, Sept. 12. E. MÜNCH, Ludwigshafen-on-the-Rhine, Ger. Assignor to Badische Anilin & Soda Fabrik, same place. 4,4'-Dibromodiphenyldisul-

fide-2,2'-dicarboxylic acid, made by treating diazotized 5-bromo-2-aminobenzoic acid with Na sulfantimoniate, the product being colorless, m. at about  $310^{\circ}$  and when freshly pptd. from its soln. being easily sol. in acetone and difficultly sol. in alc. and glacial HOAc and very difficultly sol. in b.  $H_2O$ .

U. S., 1,003,347, Sept. 12. R. EHRMANN, Charlottenburg, Ger. Assignor to J. A. Wülfing, Berlin, Ger. Digestive medicin consisting of HCl-albumin 80-90% and pepsin 10-20% formed into tablets.

U. S., 1,003,646, Sept. 19. W. OHLIGER, Detroit, Mich. Obtaining a purified fluid extract of the blood-pressor substance from suprarenal glands by extracting the glands with alc. and trichloroacetic acid, b. the ext. to ppt. inert substances, concg. the ext., pptg. the remaining inert substances by adding a salt, e. g., Pb(OAc)<sub>2</sub> or ZnCl<sub>2</sub> and eliminating the excess of the sol. salts by pptn. with a volatil reagent, e. g., H<sub>2</sub>S, all the steps of the process being carried out in the presence of a reducing agent, e. g., Na<sub>2</sub>SO<sub>2</sub>, SO<sub>2</sub> or Zn.

Brit., 26,432, Nov. 14, 1910. BADISCHE ANILIN- & SODA-FABRIK, Ludwigshafen a/R. Mfg. anthraquinone-1,2-dicarboxylic acid by treating naphthanthraquinone with an acid oxidizing agent. The anhydride is obtained by dehydrating the acid.

Brit., 29,073, Dec. 14, 1910. CHEM. FABRIK GRIESHEIM-ELEKTRON, Frankfurt a/M, Ger. In the manuf. of acetaldehyde and condensation and polymerization products thereof, by the hydration of acetylene, treating the soln. of a mercurial salt in an organic or inorganic acid with C<sub>2</sub>H<sub>2</sub> at a temp. below 70°.

Ger., 236,196, June 28, 1908. C. F. BORHRINGER & SÖHNE, Waldhof bei Mannheim. In the manuf. of acidyl derivatives of the salicylic acid of the general formula acidyl O.C., H., CO.O.C., H., COOH, either salicylosalicylic acid acidylized according to one of the known methods, or acidylsalicylic acids condensed with salicylic acid, whereby AcOAc is excepted as the condensation agent, or acidylsalicylic acid alone or in the presence of solvents, with the exception of HOAc and AcOAc, are heated for a short time in such manner as to avoid the formation of salicylide. Numerous examples are given.

Ger., 236,490, Aug. 27, 1909. Addition to 214,950, Mar. 19, 1908; cf. C. A., 4, 644. P. Koch, Berlin. Manuf. of compounds rich in sulfur (thiozonides). Instead of the alcs. and esters of the terpene series, are employed pure O-free terpenes which, upon heating with S until the evolution of H<sub>2</sub>S, admit of an increase of more than 1/2 their wt. by the addition of S. Examples are given.

Ger., 236,604, Nov. 6, 1909. FARBEN. VORM. F. BAYER & Co., Elberfeld. Process of entirely or in part replacing with hydrogen the halogen atoms in polyhalogenized aminoanthraquinones and their derivatives. By treatment of halogenaminoanthraquinones, which contain several halogen atoms, and their derivs., with reducing agents, with the exception of alkali alcoholates, the halogen ats. are successively replaced by H. The application of the process and examples are given.

Ger., 236,605, Sept. 17, 1910. H. BUER, Köln. Preparation of lecithin from the seeds of pulse and other vegetable material containing lecithin, by extracting the initial material with 96% alc. in amt. about 50 times the wt. of the ext. substance contained in the crude material, at a pressure of 0.5 to 1 atm. or with the employment of a reflux condenser at 80-90°, then allowing the alc. extraction liquid to stand for some time at the ordinary temp. until the complete sepn. of fat, cholesterol and coloring matters, then evapg. the alc. soln. which contains the bitter principles and lecithin to about 60-80%, and again allowing the soln. to stand for a long time at the ordinary temp. until the sepn. of the lecithin.

Ger., 236,769, July 3, 1910. FARBWERKE VORM. M. L. & B., Höchst a/M. Mfg. p-dialkylaminobenzyl-1-aminoanthraquinones of the general formula

 $C_0H_4$ : :  $C_0H_4$ .NH.CH<sub>2</sub>. $C_0H_4$ .NR<sub>2</sub>, by acting with HCHO in the heat on 1-amino-CO

anthraquinone in the presence of tertiary aromatic bases. Examples are given.

### 18. ACIDS, ALKALIES, SALTS AND SUNDRIES.

#### T. LYNTON BRIGGS.

Critical Consideration of the Intensive Practice in Sulfuric Acid Works. A. NEMES. Z. angew. Chem., 24, 1564.—Controversial. T. L. B.

Intensive Practice in Sulfuric Acid Manufacture. H. PETERSEN. Z. angew. Chem., 24, 1811.—Controversial.

T. L. B.

Reconstruction of Two Sulfuric Acid Factories According to the "Moritz" System. KLIPPERT. Z. angew. Chem., 24, 1345.—Two chamber systems, of wooden construction, burned down Oct. 6, 1910, were rebuilt with iron, fireproof construction of the "Moritz" system, and put in operation by March 15 and April 4, 1911. Using steam for the H<sub>2</sub>O supply with a production of about 1 lb. 50° acid for each 2.3 cu. ft. space, the consumption of 36° HNO<sub>3</sub> is 0.5-0.6%.

T. L. B.

Preparation of Borax. II. M. G. LEVI AND O. GARAVINI, Pisa, Palermo. Gazz. chim. ital., 41, I, 756-81; cf. C. A., 5, 1168.—A repetition of the expts. with dry H<sub>2</sub>BO, and NaCl at 10° intervals between 100 and 230° shows that while there is a marked increase in the velocity of reaction at 140°, as found before, it is appreciable at 120° (1% decomp. of the NaCl after 8 hrs.). The av. max. of decomp., 17%, is already reached at 170° and very quickly (15.4% after 23 min.). It seems, therefore, that the reaction begins with the formation of meta-rather than pyroboric acid and that a state of equil. which is not disturbed within certain, rather extended, limits of temp. is established when a certain amt. of Na borate is present. When borax (anhydrous) is added to the mixt., the reaction is more or less impeded, according to the amt. of borax, whereas a neutral diluent (quartz) has practically no effect. From  $500^{\circ}$  up the % of decomp. increases and Cl begins to be evolved, even when the expt. is carried out in an atm. of N. although then the amt. of Cl evolved is smaller. This indicates that the phenomenon is due both to oxidation of the HCl and to the action of the B<sub>2</sub>O<sub>3</sub> formed on the NaCl. The dissociation of HCl also doubtless plays a part; a check expt. in which dry HCl, freed from O, was passed through the Pt app. showed that dissociation began at 800° and was quite marked at 1100°. At 400°, steam has practically no influence on the velocity of the reaction, but above 500° it accelerates it greatly. Only HCl (no Cl) is formed. Even at the highest temps. (1000°), the presence of large amts, of borax sensibly lessens the reaction velocity. By reducing the time of heating and the amt. of steam to a minimum, the losses of H2BO2 and NaCl due to volatilization with the steam can be greatly lessened. The results of the earlier expts. with B<sub>2</sub>O<sub>2</sub> and NaCl have been confirmed. The reaction between H<sub>2</sub>BO<sub>3</sub> and Na<sub>2</sub>SO<sub>4</sub> is slower than that with NaCl; B<sub>2</sub>O<sub>3</sub> reacts with about the same velocity with Na<sub>2</sub>SO<sub>4</sub> as does H<sub>2</sub>BO<sub>2</sub>. The reaction begins only at 500°; SO<sub>2</sub> is evolved. Steam accelerates the reaction. Chas. A. Rouiller.

The Berrigan Continuous Bucket-chain Filter Press. J. F. BERRIGAN. Eng. News, 66, 184.—A complete description of an ingenious filter press which has many times the capacity of a screw or hydraulic press requiring the same space and h. p. The press requires 1 h. p. to drive it, handles the material at the rate of 360 bu. per hr.;

the material is pressed twice between cloths, the cake being turned automatically between pressing. The press adjusts itself automatically to the compressibility of the material, and its capacity is only limited by the speed at which it can be run safely and efficiently. 2 cuts.

E. C. LATHROP.

Potassium Permanganate. E. Schütz. Z. angew. Chem., 24, 1628-31.—The author describes fully a com. process for the manuf. of KMnO<sub>4</sub> depending on the following reactions: (a) 6MnO<sub>2</sub> + 2KClO<sub>3</sub> + 12KOH = 6K<sub>2</sub>MnO<sub>4</sub> + 2KCl + 6H<sub>2</sub>O. (b) 2KMnO<sub>4</sub> + Cl<sub>2</sub> = 2KCl + 2KMnO<sub>4</sub>. The process is carried out as follows: '(1) Fusion of crushed MnO<sub>3</sub> with KOH 50° Bé. (2) Crushing of product into fine ball meal. (3) Increasing the content of KMnO<sub>4</sub> by means of hot compressed air. (4) Dissolving the mass with dil. KOH from electrolysis and wash water. (5) Sepn. from the settled MnO<sub>2</sub>. (6) Changing of the K<sub>2</sub>MnO<sub>4</sub> into KMnO<sub>4</sub> by means of electrolysis. (7) Crystn. of salt by cooling and drying in vacuo. (8) The K<sub>2</sub>CO<sub>3</sub> formed during the process is converted into KOH. A description of the machinery and plant necessary for an output of 10,000 kg. per month is fully given together with the quantities of materials to be used. 4 cuts.

KIPPENBERGER, C.: Ueber Beziehungen der Chemie zur Rechtspflege. Leipzig: O. Spamer. 1.80 M.

LUNGE, G.: Technical Chemist's Handbook. New York: D. Van Nostrand. 283 pp., \$3.50.

LUNGE, G. AND BERL, E.: Chemisch-technische Untersuchungsmethoden. 6 Aufl. (4 Bände.) Bd. III. Berlin: 8°, 1044 pp., 22 M.

MIETHE, A.: Die Technik im 20. Jahrhundert. Die Gewinnung der Rohmaterialien. Braunschweig: G. Westermann. 8°, 397 pp., 15 M.

NAGEL, O.: The Lay-out, Design and Construction of Chemical and Metallurgical Plants. New York: O. Nagel, 50 E. 41st St. 204 pp., \$2.00.

SCHMIDT. O.: Chemie für Techniker. 2 Aufl. Stuttgart: 8°, 175 pp., 2.80 M.

WICHELHAUS, H.: Vorlesungen über chemische Technologie. 3 Aufl. Dresden: T. Steinkopff. 23 M.

- U. S., 1,001,310, Aug. 22. W. SCHULTHESS, Paris, France. Apparatus for slaking lime.
- U. S., 1,001,381, Aug. 22. D. R. FRANKLIN, New York, N. Y. Blacking for stoves and other metal surfaces, including graphite and a H<sub>2</sub>O emulsion of Co hydrate which will be decomposed by the action of heat (at about 163°) to form black Co oxide.
- U. S., 1,001,775, Aug. 29. J. SCHAMBONY, Munich, Ger. Making writing surfaces by etching a glass plate, applying to it a layer of coloring matter adapted to be burned in, combined with a glass-flux, then drawing lines on the surface with a color adapted to be burned into the glass, grinding down the lines so as not to be palpably in relief, and finally burning the covering layer and lines.
- U. S., 1,001,789, Aug. 29. L. WEISS, Munich, Ger. Making semi-opaque, vitreous compounds by cooling a fused mass composed of borax or B<sub>2</sub>O<sub>2</sub> and an oxide of Zr, Si, Sn, Ti, Th or Pb and concurrently passing steam into contact with the mass, the latter being then added to a vitreous compd.
- U. S., 1,001,836, Aug. 29. F. E. Fritz, Delmenhorst, Ger. Assignor to Soc. Bremer Linoleumwerke Delmenhorst, same place. Making plates of linoleum composition by rolling several differently prepared linoleum comps. of ordinary granules successively upon one another, forming one of the comps. prior to its being rolled in

the shape of loose sausage-like bodies, and forming another of the compds. prior to its being rolled into differently colored loosely connected masses.

- U. S., 1,001,873, Aug. 29. M. MAURAN, Niagara Falls, and J. H. MacMahon, LaSalle, N. Y. Generating oxygen by anchoring solid particles of a catalytic agent, e, g., Co oxide, in a substantially stationary body of O-yielding soln., e. g., a 20% aq. soln. of bleaching powder, maintaining the reaction until the soln. is spent and then sepg. the residue of gas-making material from the catalytic agent. The Co oxide may be incorporated with Portland cement, baked clay or plaster of Paris to form a lining for the reaction vessel or molded bodies to be suspended in the soln.
- U. S., 1,002,086, Aug. 29. F. J. SCRABLE, Pittsburgh, Pa. Making whetstones by mixing sand 4, earth 4, cement 4, paint 2 parts, H<sub>2</sub>O and oil, b., cooling, molding, tempering and polishing.
- U. S., 1,002,317, Sept. 5. G. RIGG, Palmerton, Pa. Treating sal ammoniae skimmings for the recovery of the chlorides and the production of a residue suitable for the making of spelter, by heating the skimmings to a temp. above the b. p. of ZnCl<sub>2</sub> to volatilize the ZnCl<sub>2</sub> and NH<sub>4</sub>Cl and condensing the ZnCl<sub>2</sub> to liquid form and finally recovering the NH<sub>4</sub>Cl fumes by absorption.
- U. S., 1,002,349, Sept. 5. H. P. White, Kalamazoo, Mich. Lubricating or packing material formed by crumpled sheet metal coated with oil, and powdered graphite, tale or mica folded and compacted together.
- U. S., 1,002,505, Sept. 5. T. A. Edison, Llewellyn Park, West Orange, N. J. Composition for sound records containing shellac 80% and com. tetrachloronaphthalene 20% in the form of fibrous crystals distributed throughout the shellac. Dinitrotoluene or dinitrobenzene may be used instead of tetrachloronaphthalene and a filler such as asbestos or cotton may be employed.
- U. S., 1,002,658, Sept. 5. G. E. Dunton, New York, N. Y. Treating molds used in electrotyping, to avoid accumulation of grease and black lead, by applying to the molds a mixt. of a grease-converting substance, and a conducting substance, e. g., black Pb, to render it conductive and convert the grease expressed from it into a saponaceous compd., and finally removing the latter by washing the mold with H<sub>2</sub>O.
- U. S., 1,002,659, Sept. 5. F. L. DYER, Montclair, N. J. Assignor to T. A. Edison, Inc., West Orange, N. J. Making metallic duplicate copies of sound records by rotating a matrix or mold in a Co-plating bath and depositing on the record surface of the matrix or mold an excessively thin film of Co, then subjecting the Co film to a Cu soln., e. g., CuSO<sub>4</sub>, resulting in the formation of granular, slightly adhesive cement Cu film, and then electrodepositing thereon a heavier layer of metal, e. g., Ni, and finally removing the electrodeposit, a 30-40% soln. of KCN being used if necessary to dissolve the cement-Cu and free the electrodeposited layer.
- U. S., 1,003,089, Sept. 12. A. COLLARD, Molenbeck, near Brussels, Belgium. Apparatus for recovering solvent vapors from air (s. g., ether, alc. og acetone) in the manuf. of artificial silk or smokeless powder, by absorption and distillation.
- U. S., 1,003,139, Sept. 12. J. W. Kelley, Chicago, Ill. Core-oil for binding sand, etc., consisting of a mixt. of resin oil prepared by dissolving a resinous material, (e. g., rubber resin) in Harbin oil (the latter being made by mixing a hydrocarbon oil with CaCl<sub>2</sub> [Ca(OCl)<sub>2</sub>?] and HCl) and Chinese or tung oil treated with burnt umber and mixed with Harbin oil.
- U. S., 1,003,433, Sept. 19. E. COLLETT and M. ECKARDT, Christiania, Norway. Producing ammonia by heating Ca cyanide or cyanamide with Ca(NO<sub>2</sub>)<sub>2</sub> and H<sub>2</sub>O to 120-250°, maintaining the mixture heated for some time (preferably 3-6 hrs.),

drawing off the NH<sub>2</sub> produced, neutralizing the remaining product with HNO<sub>3</sub> and drying the residue thus obtained.

- U. S., 1,003,773, Sept. 19. E. A. MERRILL, Grand Rapids, Mich. Heat-insulating material consisting of a mixt. of dry cork in relatively large granules and powdered asbestos in the interstices between the granules.
- U. S., 1,003,827, Sept. 19. F. A. TOLHURST, New York, N. Y. Drying and brightening dipped or plated metal articles by forcing hot dry air at high velocity through a mass of the articles while wet.
- U. S., 1,003,875, Sept. 19. R. CASPARI, Uerdingen, Ger. Assignor to R. Wedekind & Co. M. b. H., same place. Making granular sodium dichromate by heating a mass of the cryst. salt gradually to near its m. p. during agitation, adding such proportion of a soln. of Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> as will dry on the surface of the granules formed if desired to increase their size and removing the steam as it generates during the heating.

Brit., 15,973, July 4, 1910. BAD. ANILIN- & SODA-FABR., Ludwigshafen a/R, Ger. Mfg. ammonia from its elements by passing a mixt. of or contg. N and H under pressure over a hot catalytic agent in a metallic vessel and applying heat to the walls of the vessel which are sustaining the pressure employed.

Brit., 16,454, July 9, 1910. F. A. FREETH, Lawton Road, Gt. Crosby. Mfg. pure or nearly pure ammonium nitrate from (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> and NaNO<sub>5</sub> by stirring together at or about the temp. at which the Na<sub>x</sub>SO<sub>4</sub> pptd. by the subsequent reaction is removed from the soln., that total amt. of equiv. quantities of (NH4), SO4 and NaNO, with that quantity of a nucleus soln. which will yield, after the reaction is finished, the same ratio of NaNO, to NH, NO, in the resulting soln. at the temp, at which the Na, SO, ppt. is removed from the soln. as obtains in a soln. satd. with respect to Na<sub>2</sub>SO<sub>4</sub>, and also satd. or nearly satd. with respect to the double salt 3 AmNO, Am, SO, at that temp. when such soln. contains NaNO, and Na<sub>2</sub>SO<sub>4</sub> in the same or nearly the same ratio as obtains in the nucleus soln.; continuing the stirring, and concg. the mixt, by evapn, until the ratio of NaNO, to Na<sub>2</sub>SO, in soln. is actually the same or nearly the same as that in the nucleus soln. employed; sepg. the pptd. Na SO, from the soln. at the temp. originally decided upon, and for which the quantities and comp. of the liquors employed have been adjusted, and washing the Na<sub>2</sub>SO<sub>4</sub> to remove adherent mother liquor; adding H<sub>2</sub>O to the soln. from which the pptd. Na<sub>2</sub>SO<sub>4</sub> has been removed until the ratio of H<sub>2</sub>O to NaNO, is the same as or slightly greater than that which obtained in the original nucleus soln. employed; cooling the resulting soln. to the temp. originally decided upon for sepn. of NH<sub>4</sub>NO<sub>2</sub>, for which temp. the quantities and solns, employed have been adapted; sepg. the pptd. AmNO, from the soln. at that temp., washing it with a satd. or partly satd. soln. of AmNO, to free it from adherent mother liquor, and drying it, after which it is pure or practically pure.

Brit., 16,615, July 12, 1910. HEYLANDT-GES., Meldorferstrasse, Hamburg 30, Ger. In the liquefaction of hydrogen, employing 2 distinct but cooperating closed circuits, in 1 of which H gas is cooled to a temp. at which, on expansion a cooling effect is produced, allowed to expand to reduce its temp. from —150° to —190°, and passed through a cooler common to both circuits, in 1 direction, while in the 2nd circuit the H gas to be liquefied is compressed and purified, cooled to ord. temp., passed through the cooler in the other direction, by which the temp. is reduced to about —80°, allowed to expand for further reduction of temp. so that a part liquefies, the remainder being utilized to assist in cooling the compressed H gas passing through the cooler, and then returned to the compressor of the circuit.

Brit., 17,587, July 25, 1910. F. C. BEAUCHAMP, 8 Weston Park, Crouch End, N. For obtaining a precipitate of pure zinc hydroxide on a com. scale, the combination of

the following 4 reactions: (1) treating with H<sub>2</sub>SO<sub>4</sub> to make the metals sol. sulfates; (2) treating with lime the dissolved metals to convert them into hydroxides; (3) treating with NaOH or ammonia to make the Zn only sol.; (4) pptg. this Zn.

# 19. GLASS AND CERAMICS.

#### G. E. BARTON, A. V. BLEININGER.

The Production of Pictures and Decorations upon Glass. Anon. Diamant, 33, 872-3.—The picture or design is printed upon a sensitized gelatin layer which is developed and protected from the action of the sand blast by treatment with a soln. of a resin, glycerol and grape sugar. The glycerol preserves the moisture in the protective coating of the glass and thus prevents its hardening and wearing away under prolonged exposure to the blast. An instrument is described for producing a uniform application of the coating.

J. B. PATCH.

Proposed New Forms for Large Regenerative Glass Furnaces Using Gas as a Fuel. Hugo Knoblauch. Sprechsaal, 44, 509-12, 521-3, 536,-7. G. E. Barton.

Polishing Mediums for Use with Glass. O. PARKERT. Sprecksaal, 44, 552-3.
G. E. B.

The Tempering of Pots and Furnaces. O. Fr. Sprecksaal, 44, 483-5.

G. E. B.

The Chemistry of Sand-lime Brick. T. R. ERNEST. Univ. of Ill. Trans. Am. Ceram. Soc., 13, 648-67 (2 photomic.).—Two methods of attack were followed, chem. and microscopic. In preparing compds, for chem, analysis a high % of either sand or lime was used so that one of them would go into combination as completely as possible. Free CaO was removed by passing H.S through the soln. for several days, thus converting CaO into Ca acid sulfide; the silicate could then be washed and analyzed. CO. completely decomposed these Ca silicates. Chem. analyses of many samples made under varied conditions showed that when pure CaO and SiO, were used, the bonding material was a mixt. of hydrated Ca silicate. The major portion was doubtless CaSiO<sub>a</sub> hydrated to a greater or less extent. Silicates of a higher and lower CaO: SiO, ratio must also have been present to account for variations in analytical results. The amt. of H<sub>2</sub>O in combination was variable. Petrographic study of thin sections of sandlime bricks and of thin sections and powders of the pure CaO-SiO, series showed: (1) Isotropic crystals; (2) small, highly double refracting uniaxial crystals; both kinds were too small to be further identified. C. H. KERR.

A Method for Charting Heat Distribution in Kilns. WILLIAM CANNAN, JR. Trans. Am. Ceram. Soc., 13, 117-21. C. H. K.

Cause of Permanent Expansion in Firebrick. JOHN M. OGAN. Ohio State Univ. Trans. Am. Ceram. Soc., 13, 602-11 (8 curves).—Certain fire bricks expand permanently after being put into service. To study this phenomenon 2 clays, a flint clay and a semi-flint clay, were made up in the proportions of 100, 75, 50, 25 and 0% of each with the other. Both clays showed deformation at cone 34. The clays were ground to 16 mesh, worked by hand and briquets were burned to cones 010, 05, 3, 7 and 13. Shrinkage, porosity and true sp. gr. (by pycnometer) were detd. and plotted. Permanent expansion is "due to the formation of impervious bleb structure by volatilizing gases in the glassy matrix formed by fusion."

Resistance of Sheet Steel Enamels to Solution by Acetic Acids of Various Strengths.

ROBERT D. LANDRUM. Liske Mfg. Co., Ltd. Trans. Am. Ceram. Soc., 13, 494-501.

-Two series of tests were made upon each of 2 enamels, typical of the cheap wares.

The amts. dissolved by acids of various strengths of 1 to 100% by vol. of 99% acid were detd. The attack by the acid increased rapidly with increase in conc. from 1 to about 7% and then more slowly to the max. (in one case at 19% and the other at 15%) and then decreased. Fifty % and 1% are about equiv. in action; at 80-90% the amt. dissolved is very small. At 100% there is no attack.

C. H. K.

Porcelain Glazes. Ross C. Purdy. Ohio State Univ. Trans. Am. Ceram. Soc., 13, 550-84.—Report of class work. Glazes were tried on (1) a porcelain body biscuited at cone 4, (2) a typical stoneware clay dried to "bone dryness." In all glazes the Al<sub>2</sub>O<sub>3</sub>: SiO<sub>2</sub> ratio was 1: 6. The Al<sub>2</sub>O<sub>3</sub> varied from 0.5 to 0.8. The ratio RO: R<sub>2</sub>O varied from 8: 2 to 2: 2. The CaO: ZnO ratio varied from all CaO to all ZnO. The glost burns were carried to cones 6, 8, 10 and 12. Feldspar: In high feldspar bodies only did crazing occur. Clay: (1) Within each group increases of clay decreased fusibility, (2) with feldspar const. increases of clay decreased the heat range, (3) in each series where crazing occurred increase of clay decreased crazing. Opacity: (1) ZnO operated decidedly to produce opacity, (2) CaO had practically no effect, (3) within each series increase of clay increased opacity, (4) changes in feldspar had little or no effect, (5) variations in heat treatment had but slight effect, since within the temp. range included, all the glazes formed relatively imperfect solns.

C. H. K.

Report of the Committee on Classification of White Ware. CHARLES WEELANS AND HARRISON E. ASHLEY. Trans. Am. Ceram. Soc., 13, 102-10 (1 curve).—The committee intended to test all lines of white ware made in U. S. and some from foreign countries. The tests considered were: Body tests, (a) porosity and bulk sp. gr. (by immersion), (b) translucency (sieve test), (c) vitrification (red ink test); glaze tests, (a) craze, (b) hardness or toughness. Tests upon which work was done were: (1) Red ink test—small samples were immersed in red ink (5 g. eosin, 25 cc. NH<sub>4</sub>OH, 475 cc. H<sub>2</sub>O), for 18 hrs. and the depth of penetration noted, (2) absorption, porosity and sp. gr. were detd. by the immersion method, (3) relative translucency was obtained by noting the finest mesh screen distinguishable when the screen was held 3 in. from a 16 c. p. light and the ware held against it. Translucency was found to be inversely proportional to thickness. A small amt. of data has been accumulated.

Some Coefficient of Expansion Data on Porcelain Made from European Materials. A. S. WATTS. Trans. Am. Ceram. Soc., 13, 406-30 (1 cut, 5 curves).—Porcelain bodies were made from Norwegian feldspar, Hohenbocka sand, Zettlitz kaolin (raw and calcined), Halle clay, Lothainer clay and English china clay. Analyses of all materials and fineness tests of the non-plastic materials are given. The bodies were ground together for 2 hrs. Firing was carried to cone 15. The app. used for detg. coeff. was a glass cylinder with metal caps on both ends, and with a quartz bearing in the center of each cap. The trial piece was heated in H<sub>\*</sub>O to a const. temp., measured in length and transferred to the bearings in the glass cylinder at the same temp. The dial was then adjusted to read o. Steam was introduced into the cylinder and a const. temp. of 100° maintained. The elongation was measured and the coeff. of exp. calc. Results: (1) Coeff. was increased by substituting flint for either clay or feldspar, (2) coeff. was not greatly changed by substituting feldspar for kaolin, (3) coeff. was slightly increased by substituting ball clay for kaolin, (4) the substitution of English china clay for kaolin greatly increased the coeff., (5) coeff. was not materially changed by substituting for flint, pulv. calcined kaolin calcined to a temp. above that to which the body was burned. Discussion led to the subject of dunting. Purdy attributed it to structural weakness in the raw state caused by too little bonding power, improper handling, too close grain, laminations, structural rupture in the burned state caused by too rapid cooling, unequal vitrification, sometimes from poor drying, too much flint. Burt held that true dunting was caused largely by high coeff. of exp. Wheelans

advocated increasing flint and increasing heat treatment accordingly as the cure for dunting.

C. H. K.

Note on "Vitrification Range and Dielectric Behavior of some Porcelains." J. MINNEMAN. Ohio Insulator Co. Trans. Am. Ceram. Soc., 13, 704-5.—That time is very important in puncturing tests is shown by tests made upon the same porcelain in 2 ways: (1) By bringing the voltage up to puncturing as quickly as possible; (2) by bringing the voltage up slowly at the rate of 20 k. v. per min. until puncturing occurred. Pieces tested according to (1) averaged about 25% higher than when tested according to (2). In the data given by Bleininger and Stull (cf. C. A., 5, 364) the time factor was neglected. The discrepancies which they found may have been largely due to the influence of time.

The Construction of a Center-stack Down-draught Kiln. E. T. MONTGOMERY. Univ. of Ill. Trans. Am. Ceram. Soc., 13, 311-23 (7 figs., 2 cuts). C. H. K.

"What is the Best Form of Magnetic Separator for Removing Iron from Slip?"

Trans. Am. Ceram. Soc., 13, 639-41.—Discussion.

C. H. K.

What is the Best Substance to Provent Clay Slip from Settling in the Casting Process? What is the Effect of this Substance on the Body in Firing?" Discussion. Trans. Am. Ceram. Soc., 13, 641-5.—Purdy used Na silicate. Watts used Na silicate or Na<sub>2</sub>CO<sub>2</sub>. Mayer used both Na silicate and Na<sub>2</sub>CO<sub>2</sub>, but mixed them with the dry clay before blunging; when added to the slip they had but little effect. Weelans questioned the advizibility of using Na silicate because it increased the vitrification of the body.

C. H. K.

Cuttery Marks on Glaze. H. E. ASHLEY. Bur. of Standards. Trans. Am. Ceram. Soc., 13, 226-7.—Black marks made by cuttery on table ware are caused by the glaze becoming rough where parts have been removed by soln. In studying the impure brown boric acid in comparison with pure, white acid, it was found that a glaze containing the brown acid was much more sol., due to the presence of sulfates. English mfrs. have been more troubled than the American because the coal used in Eng. is very high in S while the natural gas used in U. S. is low in S. Where brown boric acid is used it should be replaced by the white acid using 70 parts of white acid and 15 parts borax to replace 100 parts of brown acid.

C. H. K.

The Construction of a Twelve-foot, Potter's Updraft Kiln. FRANK H. RIDDLE. Am. Sewer Pipe Co. Trans. Am. Ceram. Soc., 13, 385-6.—Complete working drawings are given.

C. H. K.

Methods Employed in Connection with the Reduction, Milling and Shipment of Quartz, Flint Rock and Silica Sand. HARRY F. SPEIR. Penna. Pulv. Co. Trans. Am. Ceram. Soc., 13, 326-35.

C. H. K.

Clay and Kaolin Mining in Europe. ARTHUR S. WATTS. Trans. Am. Ceram. Soc., 13, 228-50 (15 cuts).

C. H. K.

Some Practical Observations in the Drying of Terra Cotta. Wm. J. STEPHANI. O. W. Ketcham T. C. Co. Trans. Am. Ceram. Soc., 13, 746-50 (2 figs.).—Measurements of warpage caused by too rapid drying of 2 pieces of ashlar and 3 pieces of dental molding were made.

C. H. K.

A Study in Underglaze Colors with View of Determining the Most Insoluble and Mon-volatil Underglaze Color Composition. E. T. Montgomery. Univ. of Iil. Trans. Am. Ceram. Soc., 13, 723-30; cf. Ibid., 5, 115-21.—The object of the work was to show the truth or falsity of Seger's position in advocating the compounding of underglaze colors, whenever possible, according to the spinel formula (MgOAl<sub>2</sub>O<sub>2</sub>) using a metallic oxide with Al<sub>2</sub>O<sub>2</sub> or a metallic oxide with a metallic R<sub>2</sub>O<sub>2</sub>. Oxides and phosphates of

the common metals were compounded with Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, Al<sub>3</sub>(PO<sub>4</sub>)<sub>3</sub> and kaolin in 5 proportions, 2 above, 2 below and 1 using the spinel proportion. The oxides were: Cr<sub>2</sub>O<sub>3</sub>, Co<sub>3</sub>O<sub>4</sub>, CuO, Fe<sub>2</sub>O<sub>3</sub>, MnO<sub>2</sub> and Na<sub>2</sub>U<sub>2</sub>O<sub>7</sub> + 6 H<sub>2</sub>O. Three phosphates (Cr, Co, Fe) were used. The fritts were all calcined at cone 10, ground and washed, then mixed with 20% of the glaze to be used and fired under the glaze under 2 conditions, (1) body biscuited at cone 10 and glazed at cone 02 with a fritted glaze—representing Am. practice, (2) body biscuited at cone 05 and glazed at cone 12 with a porcelain glaze—representing European practice. Results: (1) Al<sub>2</sub>O<sub>3</sub> was the best refractory to use with the oxides, while SiO<sub>2</sub> gave better results with the phosphates; (2) varying the proportions above and below the spinel ratio gave no distinctions; (3) no evidence was obtained that volatilization of a color was influenced by departing from the spinel ratio—volatilization varied only with the kind of coloring material used; (4) the oxides were superior to the phosphates except in the case of Co; (5) the fritts stood both glost treatments without noticeable effect upon the solubility.

C. H. K.

Saggar Breakage. Discussion. Trans. Am. Ceram. Soc., 13, 645-7.—Barringer had found hand-made saggars to show greater loss due to splitting at the joint. Rectangular saggars lasted longer than round ones; average life, 5 burns. Mayer kept a record of all saggar breakage; in glost burns the loss was 10% and in biscuit burns 5%. Humphrey had obtained better life with hand-made than with machine-made saggars, perhaps because his saggars were large. In small size the machine-made saggars might be much better.

C. H. K.

Notes on Dr. Hecht's Investigation of Several Relations between Porcelain and Feldspathic Barthenware. HARRISON E. ASHLEY. Bur. Stand. Trans. Am. Ceram. Soc., 13, 259-76 (14 triax. diag.).—Hecht recorded some 1200 observations on 75 different bodies (Tonind. Zig., 1897). The data he obtained are plotted on triaxial diagrams. Clay varied from 30-85% and 3 duplicate series were made: (1) All kaolin, (2) all plastic clay, (3) 1/2 kaolin, 1/2 plastic clay. When 1/2 kaolin, 1/2 plastic clay was used the shrinkage was the mean of corresponding values for kaolin and plastic clay alone. The diagrams given show glaze fit, translucency, relative porosity, and shrinkage. The chief points shown are: (1) With Seger porcelain glaze (0.3 K, 0, 0.7 CaO, 0.5 Al, Oa 4.0 SiO<sub>2</sub>), the higher the ratio of plastic clay to kaolin the less the crazing, (2) with the high feldspar bodies the higher the quartz content the less the crazing in both porcelain and earthenware glazes, (3) as quartz in the body was increased at the expense of feldspar the porcelain glazes were better able to hold on high kaolin bodies, (4) translucency occurred in kaolin bodies at a much lower temp. than in ball clay bodies, (5) feldspathic soft porcelains were changed to earthenware by raising the content of clay substance at the expense of feldspar, (6) hard porcelains differ from earthenware chiefly C. H. K. in the temp. of burning.

The Effect of Temperature on the Dielectric Strength of Porcelain Insulators. Chester E. Henderson and George O. Weimer. Ohio State Univ. Trans. Am. Ceram. Soc., 13, 469-78.—An elec. furnace was built using the same materials as in the body to be tested. The body tested was: Tenn. ball clay No. 7, 27%; Tenn. ball clay No. 3, 6%; Georgia kaolin, 16%; flint, 16%; feldspar, 35%. The batch was wet ground, screened through 100 mesh, filter pressed and aged for 2 months. The body showed 11% fire shrinkage and 0% absorption at cone 9. Cylindrical test pieces were jiggered, dried and burned to cone 9 in 30 hrs. Puncturing tests were made at 10 temps. ranging from 24-274°. Up to 50° there was but little change, the puncturing voltage being about 60,000, above 50° it decreased rapidly to 4000 volts at about 200°.

C. H. K.

Kiln Flue Regulation According to Some Ventilation Formulas. DWIGHT T. FARNHAM. Seattle. Trans. Am. Ceram. Soc., 13, 706-22 (8 cuts). C. H. K.

An Investigation on Art Enameling on Metal. Johannes Minneman. Ohio State Univ. Trans. Am. Ceram. Soc., 13, 514-30.—Au, Ag and Cu are enameled. Throughout the expts. Cu was the metal used. The enamels were fritted, quenched in H<sub>2</sub>O, ground to 100 mesh and washed free from dust. The Cu was pickled by dipping into conc. HNO<sub>2</sub> and then into hot soda soln. and finally washing in clear H<sub>2</sub>O. Soft enamels bought from Schutt of Pforzheim, Ger., melted at 565° in 2 min. and that temp. was used in the expts. Starting with the enamel: 0.451 PbO, 0.508 K<sub>2</sub>O, 0.0413 Na<sub>2</sub>O, 0.852 SiO<sub>2</sub>, 0.0813 B<sub>2</sub>O<sub>2</sub>, variations in PbO, in SiO<sub>2</sub>: borax ratio and in Na<sub>2</sub>O: K<sub>2</sub>O ratio were studied. The results were easily fusible but not durable enamels. Increase of SiO<sub>2</sub> made them less fusible but more durable; 1.0 SiO<sub>2</sub> was too low for durability and 2.0 SiO<sub>2</sub> was too high for soft enamel. The effects of variations in the several Ro ingredients were studied. The best soft enamel flux was 0.5 PbO, 0.5 K<sub>2</sub>O, 1.5 SiO<sub>2</sub> and this compared very favorably with the com. soft flux examd. For hard enamel the most suitable was 0.5 PbO, 0.5 K<sub>4</sub>O, 2.0 SiO<sub>2</sub>.

The Clay Deposits of Washington. S. GRIJSBERK. Trans. Am. Ceram. Soc., 13, 751-64 (1 map).—The majority of the clays are tertiary or post-tertiary in age. Many deposits both developed and undeveloped are described. Analyses of 10 representative clays are given.

C. H. K.

The Commercial Clays of Utah. A. F. GREAVES-WALKER. Trans. Am. Coram. Soc., 13, 277-300 (2 maps, 9 cuts).—Utah contains but few valuable clay beds. Clays are abundant but high alkali and alk. earth contents make many of them almost valueless. Several deposits are described.

C. H. K.

The Cause and Control of the Mottling of Enamels on Metal. Homer F. Staley. Ohio State Univ. Trans. Am. Ceram. Soc., 13, 489-93.—The mottled spots are due to an Fe ppt. in the enamel coat before firing. The Fe forms are pickled in 10% 65° Bé. H<sub>2</sub>SO<sub>4</sub> (or 331/3% com. HCl). When bright they are taken out, drained roughly and quickly removed to the storage tank filled with H<sub>2</sub>O; thus the storage tank becomes a weak soln. of H<sub>2</sub>SO<sub>4</sub> and FeSO<sub>4</sub>. When the form is removed from the storage tank it is wet with dil. H<sub>2</sub>SO<sub>4</sub> and FeSO<sub>4</sub>. The enamel slip is alk. and Na<sub>2</sub>SO<sub>4</sub> and FeO.H<sub>2</sub>O are formed. The latter oxidizes rapidly to Fe<sub>2</sub>O<sub>3</sub>.H<sub>4</sub>O. The speed of drying dets. the size and to some extent the number of spots. The spotting effect of the Fe compds. is due to fluxing rather than to staining action causing the spots to fuse to a transparent glass while the rest is still opaque. Causes of poor mottling are: (1) Scales of Fe<sub>2</sub>O<sub>3</sub> on the ware, (2) too little FeO.H<sub>2</sub>O on the ware, (3) too much FeSO<sub>4</sub> on the ware, (4) storage tank H<sub>2</sub>O becoming alk., (5) enamel too acid, (6) too long or too hard firing.

Effect of Composition on the Strength of Porcelains. Lester Ogden. Ohio State Univ. Trans. Am. Ceram. Soc., 13, 395-405.—In continuing the work of E. Ogden (Ibid., 7, 370-96) porcelain bodies were made covering and extending beyond the field of workable bodies maturing at cone 10. The limits were: Clay 20-60%, feldspar 10-50%, flint, the balance. Batches were ground wet, screened through 100 mesh, filter pressed and aged for 6 wks. Three sets of trial pieces were required: (1) Discs for abrasion and absorption, (2) bars for shrinkage, (3) wedges for translucency. All were dried, biscuited to cone 010 and then burned in regular kilns to cone 10. Bodies with 40% or more flint, cracked badly. Abrasion and translucency was studied. The data showed: (1) To produce a tough porcelain, feldspar should be not less than 15%, clay not over 50% and flint not under 30%, (2) within the limits covered and with clay const. the toughness increased with increase in flint, (3) with feldspar const. toughness increased with increase in flint, (4) with flint const. toughness did not vary directly with either clay or feldspar, (5) toughness in all cases is dependent upon flint

more than upon clay or feldspar. For a strong porcelain the % should be about 35.

Chemical Porcelain. GERARD A. MURRAY. Ohio State Univ. Trans. Am. Ceram. Soc., 13, 585-96 (2 diag.).—The comp. of bodies capable of withstanding the great and sudden temp. changes of chem. lab. wares was investigated. The bodies used were the same as used by L. Ogden (cf. preceding abstr.). The limits covered were: Clay, 20-60%, feldspar 10-50%, flint the balance. The clay contained 75% of 3 kaolins and 25% of 2 ball clays. Slips were mixed to a sp. gr. of 1.6. Ten crucibles of each mixt. were cast, dried and burned to cone 10 in 40 hrs. In testing the crucibles were heated to 250° and then immersed for 3/4 of their depths in H<sub>2</sub>O at 21°. The same crucibles were again heated to 300° and plunged into H<sub>2</sub>O, then to 300, 350, 400, 450 and 500° successively. New sets were heated directly to 500° and quenched; but 4 such treatments were required before all the pieces showed cracks. Results: (1) Unglazed low feldspar bodies withstood temp. changes better than those high in feldspar; (2) with feldspar at 15% or less, 40% clay gave the best results; (3) in bodies with over 15% feldspar, high flint was better than high clay; (4) the results may be due to variation in the extent of vitrification as much as to the original variations in comp.; (5) in most cases the bodies with highest absorption were best.

C. H. K.

Experiments on Fritted Enamel Glazes at Cone 1-3. FREDERICK RHEAD. St. Louis. Trans. Am. Ceram. Soc., 13, 324-5.—Starting from a chrome-Sn-pink, various glazes suitable for majolica art ware were obtained by adding various coloring materials. The base fritt was: White Pb 57, feldspar 89, whiting 65, borax 12, flint 72, H<sub>2</sub>BO<sub>3</sub> 51, SnO<sub>2</sub> 27, Na<sub>2</sub>CO<sub>3</sub> 2.

C. H. K.

Some Effects of Gypsum on Clay. H. E. Kramm. Geol. Lab., Cornell Univ. Trans. Am. Ceram. Soc., 13, 689-703.—The action of CaCO<sub>2</sub> in clays has been studied but no data on CaSO<sub>4</sub> are available. Mixts. of washed china clay from Cornwall, Eng., with 0-50% of pure alabaster from Hillsboro, N. B., were made. Temp.-% loss in wt. curves and temp.-porosity curves were obtained, covering the range of cones 010-12. The data showed: (1) Gypsum-clay mixts., heated to 790° for 8 hrs. still retained some plasticity, (2) SO<sub>2</sub> was driven off gradually at temps. up to cone 1; above cone 1 the evolution was rapid, (3) at 950° all SO<sub>2</sub> could not be driven off; at 1100° all was liberated, if sufficient time were given, (4) in all cases vitrification changes were very sudden.

C. H. K.

The Effect of Feldspar on Kaolin in Burning. JOSEPH KEELE. Geol. Lab., Cornell Univ. Trans. Am. Ceram. Soc., 13, 731-45.—Mixts. of English china clay with microcline feldspar in varying amts. (0-40%) and varying size of grain (40-200 mesh) were made. Test bars were molded, dried, satd. with kerosene and their vols. detd. All were burned to cone 010 in 12 hrs. and finally to cones 05-12 in 3-8 hrs. Porosity decreased with the fineness of grain. Feldspar coarser than 150 mesh did not give a well vitrified body in any case, even with 40% at cone 12. With coarse-grained feldspar shrinkage decreased with increase in amt., but with 150 and 200 mesh feldspar the shrinkage increased with increase in amt. With fine-grained feldspar, fluxing effects are evident as low as cone 1. In vitrification 20% of 200 mesh feldspar was equiv. to about 40% of 40 mesh.

Plasticity of Clay. H. LE CHATELIER. von Bemmelen Gedenkboek, 163-72.—
A brief discussion of plasticity, and of its correlation with shrinkage and hardening of the ware on firing. Plasticity is attributed to the presence in the clay of very small cryst. lamellas. It is stated that plastic pastes are obtained if glauconite, or mica, is ground sufficiently long; to the lamellar form of the particles of clay is attributed its power of remaining in suspension in water.

J. JOHNSTON.

Potash Salts from Faldspar. Edward Hart. Lafayette Coll. Trans. Am. Ceram. Soc., 13, 683-8.—K occurs in feldspars in enormous quantities. Any process of recovering K commercially must produce also pure Al<sub>2</sub>O<sub>3</sub> and SiO<sub>3</sub>. H. proposes to fuse together feldspar, K<sub>2</sub>SO<sub>4</sub> and C in the mol. proportions of 4: 4: 2. A little excess of C must be used. When free from sulfide, a light green glass results. The glass is finely ground, dissolved (all but SiO<sub>2</sub>) in dil. H<sub>2</sub>SO<sub>4</sub> and the products recovered from the soln. The products are: (1) SiO<sub>3</sub>, which may be pure enough for use in pottery, (2) Al<sub>2</sub>O<sub>3</sub>, for the manuf. of Al or Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, (3) K<sub>2</sub>SO<sub>4</sub> and (4) a small amt. of KNaCO<sub>3</sub>, used for making soit soap. At present prices the operation should be profitable. In discussion, Staley gives a review of many other processes proposed

A Comparison of Commercial Ground Coats for Cast Iron Enamels. JOHN H. Cob. Ohio State Univ. Trans. Am. Ceram. Soc., 13, 531-49.—A good ground coat must have the following properties: (1) Begin to fuse at a dull red heat and remain a good glass through a wide heat range, (2) dissolve any foreign matter on the surface of the metal, (3) have a low viscosity, (4) not contain or absorb injurious gases such as SO<sub>2</sub>. Results: (1) Co<sub>2</sub>O<sub>2</sub> was of little or no value, (2) clay was unsatisfactory in fritts and when used raw was not as good as feldspar or flint in giving a good heat range, (3) for increasing heat range there was no advantage in blending the raw materials, (4) with fritts, blending greatly extended the heat range.

C. H. K.

Steel Dies as Applied to the Manufacture of Dust-pressed Wares. ALBERT E. SMITH. Elec. Porcelain Co. Trans. Am. Ceram. Soc., 13, 597-601.—A brief description is given of the materials used and the process of making and hardening steel dies.

C. H. K.

Note on the Manufacture of Silica Brick by the "Anaconda" Method. A. F. Greaves-Walker. Trans. Am. Ceram. Soc., 13, 152-6.—The "Anaconda" method was originated by the Amal. Copper Co. The raw silica is ground with 2% slaked lime in a wet pan, using more water than in the old process. Bricks are hand molded, dried on pallets on drying racks for about 3 hrs., then re-pressed and dried completely. Re-pressing does not injure the bond. The kilns are burned to cone 9. The advantages of the new method over the old pounding process are: (1) Bricks are denser, (2) the denser structure enables satisfactory burns to be made at cone 9, while the old process required cone 13-16, (3) the bricks are easier to cut. Considering the decreased losses in the new process there is little, if any, difference in cost.

C. H. K.

Notes on Chrome-Alumina Colors. A. S. WATTS. Trans. Am. Ceram. Soc., 13, 301-5.-Marquardt of the Royal Porcelain Works had exhibited chrome-alumina colors, which by daylight are more purple than pink. To study this field, Watts made 3 series of tests: (1) Al(OH), and Cr,O, ground together and calcined, (2) Al(OH), Cr<sub>2</sub>O<sub>2</sub> and H<sub>2</sub>BO<sub>2</sub> ground together and calcined, (3) Al<sub>2</sub>(SO<sub>4</sub>)<sub>2</sub> and K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> dissolved in HCl and pptd. together by NH4OH in the presence of EtOH. In (1) a pinkish gray at cone 18 and purplish gray at cone 22 were obtained in a mixt. containing Al(OH), 93.48%, Cr<sub>2</sub>O<sub>2</sub> 6.52. In (2) a pinkish gray at cone 18 and purplish gray at cone 22 were obtained with Al(OH), 85.42%, Cr<sub>2</sub>O<sub>2</sub> 6.25, H<sub>2</sub>BO<sub>2</sub> 8.33. In (3) a pinkish gray at cone 18 and lavender-gray at cone 22 were obtained with Al(OH), 84.2%, Cr(OH), 15.8. Under several glazes at cone 14 these 3 stains gave good colors. Stains calcined at cone 18 were more pink than those at cone 22. Stains from (2) gave the best color; (1) was next, while those from (3) gave very pale colors. With the addition of ZnO, pink colors were also obtained. With TiO, a purplish brown resulted. For firing at cones 12-16 these stains are practical. Under soft white ware glazes they are unsatisfactory. C. H. K.

Influence of Clay, Feldspar and Flint on Coefficient of Expansion of Certain White

Ware Mixtures. Biscuited at Cone 10. Ross C. PURDY AND AMOS P. POTTS. Ohio State Univ. Trans. Am. Ceram. Soc., 13, 431-68.—The work of previous investigators is reviewed. Bodies used in these expts. were the same as those used by Ogden. Trial pieces were pressed into plaster molds, dried, placed on sand in saggars and burned to cone 10 in 40 hrs. For detns. of coeff., trials were heated in a small elec. furnace made of asbestos lumber and lined with oxychloride cement and asbestos. Readings were taken at 100, 200, 300, 400 and 500°. The chief source of error was in the structural defects of the trial pieces. The temp, may have varied ±5°. Coeff. data were shown by a plaster model built on a triaxial diagram base. All bodies containing 25-30% feldspar, showed low coeffs. The minimum coeff, was that of a body containing 30% feldspar, 30% flint and 40% clay. By keeping feldspar and clay in this same ratio and increasing flint it was found: (1) The 1st 5% addition of flint caused an abrupt rise in the coeff. of exp. from 0.0000041 to 0.0000052, (2) the 2nd 5% caused less marked increase, (3) the 3rd 5% caused slight decrease, (4) further additions up to 60% caused increases. The highest coeff. was obtained with the highest % flint and accompanying it came shivering of the glaze in accordance with Seger's rule, But increases of flint did not always increase the coeff. and the ability of the given giaze to stand without crazing could not be found to be a function of any particular coeff. of the body. The coeff. of the body was neither the sole nor the most effective factor in detg. glaze fit. The coeff. was more a function of structure than of mineral comp. No permanent set was obtained in trial pieces. No relation was found between the coeff. of exp. of bodies and the ability of small crucibles to stand quenching from a red heat. C. H. K.

Notes on Roofing Tiles and their Manufacture. Charles Stolp. Trans. Am. Ceram. Soc., 13, 306-10. C. H. K.

Seger's Rules for Correction of Glaze Defects by Changing the Body. Ross C. PURDY. Ohio State Univ. Trans. Am. Ceram. Soc., 13, 157-209 (6 diag.).—White ware bodies, varying from 0 to 100% in feldspar, clay and flint and covering the entire area of the triaxial diagram were made. Canadian feldspar, Ohio silica flint and a mixt. of ball clays and china clays were used. Biscuit burns were carried to cone. 1, 3, 5, 7, 9 and 11 in 30-60 hrs. All biscuits were glazed at cone 3 with the following fritted glaze: 0.3 PbO, 0.05 Na<sub>2</sub>O, 0.25 K<sub>2</sub>O, 0.40 CaO, 0.35 Al<sub>2</sub>O<sub>2</sub>, 2.49 SiO<sub>2</sub>, 0.47 B<sub>2</sub>O<sub>3</sub>. The results are shown on triaxial diagrams, one for each biscuit burn. The conclusions were: (1) Shivering occurred in all high flint bodies, the area decreasing rapidly as the biscuit burn increased from cone 5 to 11; (2) crazing was produced by low flint and high clay whatever the feldspar content; (3) high feldspar bodies crazed when biscuited at cones 1 and 3 but at higher biscuit burns crazing ceased; (4) with this glaze and with flint 30-50% and biscuited at cone 5 or above the feldspar: clay ratio may vary widely without causing crazing or shivering; (5) "Seger's rule concerning the effect of feldspar applies to but few of the practical bodies unless it be understood to apply only to substitution of feldspar for flint or vice versa;" (6) between flint : clay ratios of 7:3 and 3:3 Seger's rules do not apply—in other ratios they hold; (7) as the biscuit burn increased to cone 7 the craze area decreased—beyond that cone it increased again; (8) results will vary with the type of glaze used; (9) Seger's rules cannot be applied rigidly; (10) higher flint content in white ware bodies is recommended. Discussion: Binns held that feldspar in the clays must be considered and that the comp. as shown by rational analysis (calc. from ultimate analysis) was essential. Burt placed much dependence upon fineness of grain. He criticized P.'s methods and believed that his conclusions were too broad and sweeping. Burt's experience had shown much narrower range between craze and shiver than Purdy found. Maddock attributed all crazing to body defects and variations in firing. Weelans was opposed to the use of 40% flint in an earthenware body. P. believed that change of the "china clay to ball clay" ratio would change the results. He attacked both the H<sub>2</sub>SO<sub>4</sub> digestion and the calc. rational analysis. C. H. K.

Experiments on the Drying of Certain Tertiary Clays. Edward Orton, Jr. Ohio State Univ. Trans. Am. Ceram. Soc., 13, 765-91.—The clays investigated were either of late Cretaceous or of early Tertiary age. Twelve samples were taken comprizing shales, buff-burning fire clays and red-burning fire clays. Ordinary tests showed great stickiness, excessive drying shrinkage, reasonable burning shrinkage, good color, hardness, vitrification, etc. To overcome the drying shrinkage the following tests were made: (1) Adding non-plastic materials; (2) using chem. coagulants; (3) pre-heating. The addition of sand, burnt clay and saw-dust were all unsuccessful in producing a workable mass. The use, as chem. coagulants, of NaOH, Na<sub>2</sub>CO<sub>2</sub>, H<sub>2</sub>SO<sub>4</sub> and straw were equally unsuccessful. Pre-heating at 100°, 150° and at 200° had no appreciable effects. Pre-heating at 300° decreased the stickiness and improved the drying but was far from correcting it. Pre-heating at 330°, 400° and at 450° gave improvement but inconsistent results. By pre-heating at 480°, 580-625° and at 470-490° in 3 different runs, the drying was improved but was still very inconsistent, probably because of irregularities in heating. A mixt, of the shales heated to almost a red heat gave a mass that was only slightly plastic until ground with water and of which a full-sized brick would dry in 3-4 hrs. without a flaw. A second rotary furnace was set up in which the clay was stirred by rotation but from which it did not escape. Heating to 450-510° gave satisfactory results with all samples, the time varying with different samples. The time was the critical point, in some cases the margin between good and bad results being as narrow as 5 min. Pre-heating at 450-510° produces change of color, decrease in plasticity and elimination of drying cracks. Analysis of the mixt. of shales showed changes from the original condition (a) to the pre-heated condition (b): C, (a) 1.81, (b) 0.09; total H<sub>2</sub>O, (a) 11.25, (b) 7.83; H<sub>2</sub>O expelled at 105°, (a) 7.35, (b) 4.73; H<sub>2</sub>O expelled at red heat, (a) 3.90, (b) 3.10. The base of these clays may be bentonite, an Al silicate of higher H<sub>2</sub>O content than kaolinite. Bentonite is of wide occurrence through this district. The main facts brought out are: (1) That the pre-heating to be effective must be at much higher temps. than have previously been used, (2) that a loss of combined H<sub>2</sub>O is a necessary step in correcting the drying troubles of these clays, (3) that the elimination of hydrocarbons is coincident with successful treatment if not a necessary precedent thereto. C. H. K.

Relative Solubility of Clay and Flint in Feldspar. Ross D. Purdy. Ohio State Univ. Trans. Am. Ceram. Soc., 13, 479-88.—Purdy holds that flint is more sol. than clay in feldspar and bases his opinion upon the following points: (1) No eutectic has been found between clay and feldspar while between flint and feldspar one is known to exist; (2) a study of 109 bodies covering wide variations showed that "translucency decreased with increase of clay and that lines of equi-translucency radiated from the flint apex of the triaxial diagram, swinging more and more toward the feldspar corner as the biscuit heat treatment increased in intensity;" (3) in the Cramer warpage test the max. warpage occurred in the eutectic area, flint additions affecting it but little while clay additions greatly decreased the tendency to warp; (4) porosity decreased more rapidly in high flint than in high clay bodies; (5) Simonis' curves show that mixts. of equi-refractoriness can contain more flint than spar. In feldspar-flint mixts. the eutectic is the solvent; in feldspar-clay mixts. the feldspar is the solvent. C. H. K.

The Function of Time on the Vitrification of a Shale. A. V. BLEININGER AND T. L. Boys. Univ. of Ill. Trans. Am. Ceram. Soc., 13, 387-94.—The authors made tests upon a high-grade paving brick shale from Streator, Ill. The av. porosity at 650° was 37.4%. Cylinders (16-19 g.) were burned at 650° and subsequently heated

in a small Pt resistance furnace to 900, 950, 1000, 1050, 1100, 1150 and 1200°. The total variation in rate of heating during all 42 furnace runs was from 16 to 2.05° per min. Below 1100° the porosity became practically constant at relatively high porosities and the data were, therefore, omitted. The 1100° and 1150° porosity-time curves showed a distinct slope. The 1100° curve would, theoretically, meet the min. porosity of the 1200° curve after 1360 min.; the 1150° curve would require 970 min. If expts. show these curves to continue and to meet as indicated, then heating at a uniform rate to 1100° in 1360 min. would be equiv. in affect to heating at a uniform rate to 1200° in 585 min. Further work will be done.

The Oxygen Ratio. H. H. STEPHENSON. Trans. Am. Ceram. Soc., 13, 612-7.

—In the study of glazes, variations due to the body should be eliminated. This can be done by studying cylinders of the glaze fused in a metal, refractory glass or fused SiO<sub>2</sub> vessel. Only the inner core should be used. The O ratio is criticized as "pernicious," "entirely arbitrary" and meaningless. The classification of Al<sub>2</sub>O<sub>3</sub> as a base is challenged. In discussion, Orton defended the use of the O ratio as a tool capable of showing, in simple form, comparisons between bodies of the same general type; as, for instance, in a restricted field of silicate bodies within which the bases are const. and only the acid allowed to vary. The more complex the silicates the greater is the necessity of having a simple mode of correlating compds. The O ratio has been used to advantage by mineralogists and metallurgists.

C. H. K.

Fluxes and Fusion. Ross C. Purdy. Ohio State Univ. Trans. Am. Ceram. Soc., 13, 75-85.—A definition for m. p. flux and for fusion is given. Three classes of fluxes are described: (1) New compds. resulting from chem. reactions in the mass; (2) eutectics between components, no chem. reaction taking place; (3) the more fusible component in an isomorphous mixt. As an excellent example of (2) the eutectic between SiO<sub>2</sub> and CaSiO<sub>2</sub> is cited. Any component present in excess of eutectic proportions is refractory in the given case whether it be of high or of low m. p. The basic oxides, as such, are seldom fluxes.

C. H. K.

Some Observations on the Qualities of Paving Bricks. EDWARD ORTON, JR. Ohio State Univ. Trans. Am. Ceram. Soc., 13, 792-820.—Studies were made of (1) differences in rattler loss due to position of the bricks in the kiln; (2) variations due to structural differences in apparently good bricks of the same brand; (3) comparisons of standard brands. Conclusions: (1) The middle bricks were best, showing medium porosity and lowest rattler loss, the top bricks showed lower porosity and greater rattler loss, the bottom bricks the reverse. The av. variation due to irregularities in burning treatment need not exceed 2-3%. (2) Av. variations due to structural differences did not exceed 2-3%. (3) In tests of 10 standard brands, 7 showed less than 20% av. rattler loss. A variation of 3-5% in rattler loss must be expected. No definit relationship has yet been established between rattler loss and the value of bricks in use.

The Advantages of "Souring" Clays. P. ROHLAND. Deut. Toepfer Ziegler Ztg.; through Brit. Clayworker, 20, 101-2.—The practice of souring or aging is of Chinese origin. The advantages are: (1) Increase in plasticity; (2) uniform distribution of H<sub>2</sub>O. To account for (1) the following theories have been offered: (a) Bacterial growth; (b) decomp. of FeS<sub>2</sub>; (c) reduction of sulfates; (d) decomp. of feldspar by CO<sub>2</sub> and H<sub>2</sub>O. Seger held that (1) was due to hydrolysis of feldspars with the resultant formation of NaOH (in soln.) and colloidal forms of SiO<sub>2</sub> and of the hydroxides of Al and Fe and to the decomp. of organic matter by bacteria and ferments. The acids produced more than neutralize the alkali set free and therefore coagulate the colloidal substances. In aging the cellar should be cool. Rohland found that the addition of 2% tannic acid to Zettlitz kaolin doubled the tensil strength; dextrin trebled it. Starch paste,

NaOH and especially colloidal Al(OH), produced profound effects. Clays that are too plastic may be reduced to good working condition by adding alkalis or basic organic materials and storing in a warm place.

C. H. K.

Melting-point and Deformation Eutectics. H. F. STALEY. Ohio State Univ. Trans. Am. Ceram. Soc., 13, 668-82.—M. p. eutectic is defined as the mixt. of a given set of minerals which requires the lowest temp. to change the materials from the cryst. to the amorphous state. This is the eutectic ordinarily considered. "Deformation eutectic may be defined as that mixt. which requires the least heat treatment to cause fluidity sufficient to result in deformation of the mixt." The m. p. curve may lie above, upon or below the "deformation" curve according to the relative effects of the following: (1) The greater the fluidity of the melted part the greater the tendency to deform, (2) the greater the amt. of the m. p. eutectic the greater the tendency to deform, (3) the more refractory the excess components the greater the tendency to deform before complete melting. The application of these principles is made to (a) vitrification of ceramic bodies, (b) softening of glass and (c) fritting of glazes.

C. H. K.

The Measurement of Color of White Ware and White Ware Materials. Harrison R. Ashley. Bur. of Standards. (Edited by P. G. Nutting.) Trans. Am. Ceram. Soc., 13, 111-6 (1 chart).—White wares differ in color in either hue (quality) or shade (quantity) of the light reflected, or in both. Color standards to be satisfactory must measure the intensity and quality of the light reflected. The eye can detect differences in light intensity of about 1 1/2%. Measurement of color by direct comparison with color standard trial pieces is open to serious objection (see the original). "Ives universal colorimeter" (cf. C. A., 1, 2963; 2, 610) is not sufficiently sensitive. The Lovibond tintometer has given very discrepant results (cf. C. A., 4, 526) but its sensitiveness makes it promising. Dr. Nutting made measurements upon Ashley's buff and gray color series using the Marten photometer. The results are plotted in curve form. Measurements were also made upon various samples of white ware, the results ranging from 68 to 93 when compared to Ashley's standard white as 100. C. H. K.

Some Experiments on the Color of Soda-lime Glass. K. TAKAHASHI. Hazel-Atlas Glass Co. Trans. Am. Ceram. Soc., 13, 251-8.—The green color of glass is usually ascribed to the presence of Fe. MnO, is the common decolorizer. Its action may be (1) chem., oxidizing Fe compds. to the ferric state and making their color less noticeable, or (2) physical, the pink from Mn and green from Fe being complementary colors. Expts. were made to show the influence of various materials on color. The glass batch was sand 200, soda ash 80, burnt lime 23 lbs. The sand contained 99% SiO. 0.06% Fe.O.; soda ash, 99% Na, CO.; burnt lime 55% CaO, 38% MgO, 0.1% Fe,O. MnO. is usually added in slight excess and "powder blue" used to mask the pink color. A batch using 20 oz. MnO, 1/2 oz. "powder blue" and varying amts. of As,O, gave good color. With no As,O, the color was quite green. With 2 oz. As,O, the glass was colorless-larger amts. were no better. Above 10 lbs., As,O, gave a pale yellow color. "Needle Sb" gave about the same effects as As,O3, but 5 lbs. gave a fairly dense greenish vellow color. The use of niter in place of Na<sub>2</sub>CO<sub>2</sub> gave much better color when dirty cullet was used. Using fluorspar, the greater the amt., the better the color; the metal plained more easily. Feldspar increased the green color "due to the presence of a large amt. of Al<sub>2</sub>O<sub>2</sub> which seems to make the glass more susceptible to reducing actions." Kaolin and free Al<sub>2</sub>O<sub>2</sub> had like effects. The MgO content of the lime had no effect upon the color but low MgO glass plained at a lower temp. and a shorter time. C. H. K.

Range in Composition of Glazed White Ware Bodies Made from Clay, Feldepar and Fint. Ross C. Purdy. Ohio State Univ. Trans. Am. Ceram. Soc., 13, 36-

96.—The industries using white ware bodies are (a) porcelain, china, belleek, (b) general white ware and decorative ware, (c) majolica, (d) wall tile and dry-pressed elec. insulators. In all white ware bodies there is but little variation from the old formula: clay 12, flint 9, feldspar 4. The results of investigations made by Hecht, Weelans, Purdy and students at Ohio State Univ. are plotted on triaxial diagrams and discussed. The conclusions are that (1) color, translucency and glaze fit are the important criteria upon which to base a classification of white ware bodies; relative absorption is misleading; (2) the range of body comp. is narrow; the differences are chiefly in the combination of brands of clay, feldspar, etc.; (3) the correct heat treatments in bisque and glost burns and the comp. and prep. of the glaze are equally important. C. H. K.

Porcelain Kilns Using Generator Gas from Lignite Briquets. W. Stampe. Sprecksool, 44, 247-9.—The av. comp. of Czerny-Deidesheimer generator gas obtained from lignite using a steam jet is as follows: 4.7% CO<sub>2</sub>, 0.17% O, 84% C<sub>2</sub>H<sub>4</sub>, 3.17% CH<sub>4</sub>, 11.57% H<sub>2</sub> and 31.2% CO. The calorific value was about 1751 cal. per cm. The mean temp. of the gas was 170-200°. The air for combustion is drawn through the kiln grates by natural draft and the gas is introduced through an opening above the grate. A coal fire is maintained until the ignition temp. is reached. A recuperator system to utilize the waste gases is advizable. The firing of the porcelain to cone 14 may be completed in 13 hrs. 35% of fuel may be saved. No smoke is produced.

A. V. Bleininger.

The Relation between the Artist and the Chemist in Ceramic Manufacture. Herman C. Mueller. Trans. Am. Ceram. Soc., 13, 97-101.—A plea for closer cooperation between the chemist and artist, especially in the development of ceramic societies.

C. H. KERR.

The Behavior of Fire Bricks under Load Conditions. A. V. BLEININGER AND G. H. Brown. Bur. Stand., Pittsburgh. Trans. Am. Ceram. Soc., 13, 210-25 (7 illus., 3 tables, 1 curve).—Last yr. bricks were tested at 1300° under a load of 75 lbs. sq. in. (C. A., 5, 179); this yr. tests were made at 1350° under a load of 50 lbs. sq. in. Both tests condemn poor materials with certainty, but the 1350°-50 lb. test is the more sensitive and differentiates the more sharply. In the latter test, pressure effects predominate while in the former, softening due to heat is more pronounced. The load test is of value not only in testing load-carrying capacity but also in indicating actual refractory value, for in practically all uses the bricks are subjected to load. A No. 1 clay refractory should meet the conditions given, (1) stand the 1350°-50 lb. test, (2) soften not below cone 31, (3) show an RO content in no case exceeding 0.24. Analyses of fire bricks and the results of tests are given. The relation between comp. and the ability to withstand the 1350°-50 lb. test is shown graphically. No fire bricks with over 0.225 RO stood the tests, and with SiO, increasing above the Al<sub>2</sub>O<sub>2</sub>: 2 SiO<sub>2</sub> ratio, the RO becomes much more effective in reducing the refractory value. It has again been shown that the refractory nature of lower grade bonding clays is greatly improved by grinding them intimately with flint clay. Bricks were made of (a) No 2 fire clay alone and (b) a mixt. of 70% No. 2 fire clay and 30% flint clay ground together to pass 20 mesh. All were burnt to cone 12. Tests at 1300° and with a load of 50 lbs, sq. in. showed softening and distortion in bricks (a) while bricks (b) stood the test. The 1350°-50 lb. test is recommended for the testing of high-grade refractories.

A Review of the Theories of the Origin of White Residual Kaolins. HEINRICH RIES. Cornell Univ. Trans. Am. Ceram. Soc., 13, 51-74; cf. C. A., 2, 1545, 2665; 3, 161, 2663.—R. concludes that kaolin may be formed after reviewing the theories, (1) weathering, (2) volcanic waters and vapors, or (3) acidulated waters from coal beds and swamps. All of the workable kaolin deposits of U. S. and probably many

of those of Central Europe were formed by the work of surface waters entering either directly or after filtering through swamps or peat beds.

C. H. K.

Theories Concerning the Plasticity of Clays. C. SNYDER. Oklahoma Geol. Survey. *Mining Sci.*, 64, 106.

A. V. B.

The Chemical Properties of Clays. L. C. SNYDER. Oklahoma Geol. Survey.

Mining Sci., 64, 81.

A. V. B.

DJAKONOW, D. AND LERMANTOFF, W.: Die Bearbeitung des Glases auf dem Blasstische. 2 Aufl. Berlin: R. Friedländer. 8°, 196 pp., 7 M.

DRALLE, R.: Die Glasfabrikation. München: R. Oldenbourg. 40 M.

RIEKE, R.: Das Porzellan. Hannover: Max Jänecke. 8°, 191 pp., 4.60 M.

- U. S., 1,001,413, Aug. 22. G. KEPPELER, Darmstadt, and ALBERT SPANGEN-BERG, Merzig, Ger. Treating clay, kaolin and ceramic masses to facilitate molding or casting them, by adding thereto NH<sub>2</sub> and humic acid.
- U. S., 1,001,511, Aug. 22. J. H. DANVER, Beaver, Pa. Making mottled or colored enamel by fusing 2 or more portions of coloring matter of different colors with different batches of glass, grinding the resulting materials separately and mixing 2 or more of them with white or lighter colored enamel.
  - U. S., 1,002,375, Sept. 5. H. L. DIXON, Knoxville, Pa. Glass furnace.
- U. S., 1,002,954, Sept. 12. J. I. Arbogast, Pittsburgh, Pa. Removing the thread or string projecting from a gathered batch of glass by quickly passing it close up to the bottom of the holder through a gas flame or elec. arc to sever it flush with the bottom of the holder without changing the consistence or temp of the lower end of the batch.
- U. S., 1,002,955, Sept. 12. J. I. Arbogast, Pittsburgh, Pa. Making glass case goods by heating the outlet end of a blow pipe to a temp. capable of m. a mass of powdered glass, then placing the pipe with the heated end up into a core forming mold, then forming within the mold directly upon and by the heated end of the pipe from a mass of powdered glass a batch foundation spaced throughout from the walls of the mold, surrounding this foundation with plastic glass of different character to complete the batch, then forcing air through the pipe to form the batch into a core, then placing the pipe with the core thereon in a blow mold and blowing to complete the article.
- U. S., 1,002,956, Sept. 12. *Idem*. **Making glass case goods** by superimposing 2 masses of plastic glass of different character upon the heated outlet end of a blowpipe in a mold and uniting into a unitary core by blowing through the pipe, and finally blowing through the pipe to complete the article.
- U. S., 1,002,993, Sept. 12. F. HÜSERICH, Elberfeld, Ger. Making imitation stained glass by embossing a sheet of paper, coating one face with a layer of transparent material, such as varnish, thin enough to follow its contour and coating the other face with transparent material thick enough to fill all the depressions.
- U. S., 1,003,271, Sept. 12. H. A. KENT, Bounds Green, England. Assignor to Silica Syndicate Ltd., London, England. Making silica-glass by passing scattered particles of SiO<sub>2</sub> through a heated zone onto a movable heated receiving body, the particles being subjected to a temp. sufficient to sinter them by this single operation into a homogeneous fused mass adapted to be at once manipulated into the desired shape.

## 20. CEMENT, AND OTHER BUILDING MATERIALS.

#### C. N. WILEY.

Method for Rapidly Determining Iron Oxide in Cement. A. GOLUBINZEFF. Chem. Ztg., 35, 961-2.-Moisten 10 g. cement in an Erlenmeyer flask with 10 cc. H<sub>2</sub>O, let stand 10 min., add 75 cc. H<sub>2</sub>O, and then, slowly and with shaking, add 100 cc. N HCl. Boil to complete soln., reduce with H,S or SO, drive off excess reducing agent by b. while passing a current of CO, through, add MnSO, soln. and titrate with KMnO, PERCY H. WALKER.

Cement as an Iron Preservative. Anon. Can. Eng., 21, 317.—Tests are to be made by the Panama Canal Com. to det. value as a preservative of cement mortar applied to Fe plates by the "cement gun."

Corrosion of Iron and Steel in Concrete. EDIT. Engineer, 112, 259.—Review. A. J. P.

Railway Sleepers of Reinforced Concrete and Asbestos. Anon. Concrete Constr. Eng., 6, 719-20.—A Bavarian R. R. sleeper composed of asbestos fibers soaked in water and sat. with cement. The setting is much slower than that of concrete and much heat is evolved. After setting it may be drilled, nailed and hammered like wood.

A. J. PHILLIPS.

The Condition of Concrete Structure in Boston Harbor. S. C. WILLIS. Eng. Record, 64, 371-2.—The only structure not in a disintegrating cond. is a 1:2:4 test pile driver in 1909. It was a very wet mix with no mortar face and was aged 21 days before driving. The rest are in various stages of disintegration due to frost, shock of floating objects, dirty and oily sea water, poor bond between face and body of concrete and use of dry mixes. The fact that most of the disintegration occurs between high and low water indicates that frost is a prime cause and the following are recommended for proper sea water construction. (1) A fairly rich mix, not less than 1:2:4. (2) The concrete should be aged before immersion. (3) The work should be continuous where possible. (4) Iron ore cement should be used where chem. action is anticipated. A. J. PHILLIPS.

Methods of Testing Construction Materials. C. ZIELINSEI. Ciment, 16, 147-9. -Cements rapidly lose their cohesive force as the % of mixing water is increased. With regard to the sand for mortars the cohesive force decreases as the quantity of foreign material in the sand increases and the harder and sharper the sand grains the greater the resistance of the mortar. Mortars made from sand containing grains of varied size are better than those made with sand of the same sized grains. Max. strength is secured when the vol. of the mortar is 1.10 times the vol. of the voids of the sand. A. J. PHILLIPS.

Dampness in Buildings and a New Method of Prevention. M. A. KNAPEN. Ciment, 16, 154-5.

Sewer Gas and Concrete. METCALF AND EDDY. Eng. Record, 64, 408 .- In a report to the City Engineer of New Bedford, the lining of a concrete sewer with hard burnt brick is recommended whenever salt water is mixed in the sewage, as a larger amt. of H<sub>2</sub>S is there expected. An alternative is to use richer concrete and a shell 1-2 in. thick. LANGDON PEARSE.

Howard Standard Tests for Asphalt Cements for Sheet Asphalt Pavements. J. W. HOWARD. Eng. Contracting, 36, 352-4.—From lab. tests of several thousand cements H. prepared a chart showing the characteristics of all the available refined asphalts and fluxes. A column of limits is given as follows: Sol. in CS, 100-65%; organic not bitumen, o-35%; mineral not bitumen, o-35%; petrolene, 80-70%; asphaltene, 30-20%; bit. sol. in ether, 86-78%; sp. gr., 0.993-1.28; m. p., 140-170° F; chewing test good; flask-open cup, 400-600° F; ductility at 77°, 10-100; penetration at 32° No. 2 needle, 100 g. 5 sec., 0.11-0.02; same at 77°, 0.50-0.75; same at 100°, 1.35-2.70; pliability on breaking glass slight; evap. at 212° for 1 hr. 0-0.8%; same at 325° F. for 5 hrs., 0.2-2.5%, hardening of residues compared with penetration of a. c. test, 50-0; water effect immersed 1 wk. grayish, —0; same 30 days grayish, 0; adhesion at 32° above 150, at 77° 1 sq. in sec. above 25°; compression at 32° above 400 lbs. per sq. in., at 77° above 110. These do not apply to bitumen-poured and stone mixt. pavements.

A. J. Phillips.

"Properties and Uses of Douglas Fir." McGARVEY CLINE AND J. B. KNAPP. U. S. Forest Service, Bull. 88; Eng. News, 66, 380-3.—Tables and diagrams showing tests are given as well as some grading rules and specifications now in use.

C. N. WILEY.

The Problem of Waterproofing. RALPH C. DAVISON. Cement Eng. News, 23, 307-10.—The author describes the various methods of waterproofing concrete in one of the best articles which has appeared on this subject.

C. N. WILEY.

Causes of some Failures of Creosoted Wood. Block Paving in Chicago. C. K. MOHLER. Eng. News, 66, 365.—M. points out some defects in this type of pavement and recommends that certain precautions be taken (see original). Probably the best method of bedding is the use of Portland cement with the sand bed. C. N. W.

The Development of Street Pavements and Road Construction as Shown by Patents Granted by U. S. Government. Anon. Eng. Contracting, 36, 256-60, 278-81, 321-8.—A list of patents with illustrations.

A. J. Phillips.

Improved Instruments for the Physical Testing of Bituminous Materials. H. ABRAHAM. Good Roads, 13, 127-30.—Paraffin oil has been replaced by castor oil in the fuse meter and the internal friction of the consistometer has been reduced by simplifying its construction; the consistency of bituminous materials is now expressed as the cube root of the no. of g. which must be applied to a 100 sq. mm. plunger to cause it to displace the substance at the rate of 1 cm. per min. A mold of minimum cross section has replaced the old mold. The hardness of bit. substances tests between 2 and 5 units at their fusion pt. A new figure—the susceptibility factor—expresses the susceptibility to changes in temp. "S" factor = (Hd. at 32° F.) — (Hd. at 115° F.)/fusion pt. × 100. Tar pitches have a factor over 100, straight asphalts between 50-100 and blown asphalts and fatty acid pitches below 50. The factor is not dependent on the hardness or fusing pt. but is fairly constant for all bit. substances obtained from the same origin and produced by the same gen. process. No relation appears to exist between hardness and tensil strength of the bit. materials. In petroleum asphalts the tensil strength seems to be reduced if distillation is carried beyond the hard, brittle stage. Excessive blowing produces the same result but to a less degree. With bit. substances from the same source and manufacture there appears no relation between ductility and hardness or tensil strength. In each substance there is a certain temp. at which ductility is at a max. This temp. however bears no relation to the fusing pt. A. J. PHILLIPS.

The Ohio State Experimental Road. Anon. Can. Engineer, 21, 340-4.—A report of inspection of 17 sections of road laid in 1909: Glutin, somewhat dusty; stand. macadam asphalt binder, good; "pioneer" asphalt, hard and dustless; "Jarvia X," dusty; "Jarvia B," better than "Jarvia X;" liquid asphalt, hard and smooth; "ugite"—a water-gas tar with no free C, good; Fairfield asphalt cement, good; "asphaltoilene," good; Wadsworth macadam, good; "carbo-via," coal tar product, good, but slightly dusty; concrete macadam, water bound macadam with cement in

top course screenings, very dusty—addition of cement has not been beneficial; "jaroid," liquid coal tar pitch, much bleeding and uneven surfaces; "petrolithic" pavement, soft, very bad condition; limestone concrete, cracked and wearing at joints where batches were joined; gravel concrete, harder than preceding; waterbound macadam, very dusty.

A. J. Phillips.

# Chemistry of Sand-lime Brick (ERNEST). 19.

- U. S., 1,001,582, Aug. 22. C. ELLIS, Larchmont, N. Y. Making cement clinker by calcining cement material in a rotary inclined kiln section by heated flame gases and admixed aq. vapor, adding a limited amt. of H<sub>2</sub>O to disintegrate the calcined material and form H<sub>2</sub>O vapor, transferring the latter to the kiln to aid in calcination, regrinding the calcined material and clinkering the reground material in a rotary-kiln, the calcining kiln section drawing flame gases from the rotary clinkering kiln.
- U. S., 1,001,657, Aug. 29. J. M. Long, Chicago, Ill. Composition for preserving wood, composed of NaCl 12 oz., CaSO<sub>4</sub> 1 oz., ZnSO<sub>4</sub> 1 oz., CuSO<sub>4</sub> 0.75 oz., FeSO<sub>4</sub> 0.25 oz. and H<sub>2</sub>O<sub>3</sub> gals.
- U. S., 1,001,695, Aug. 29. A. E. Schutte, Newton, Mass. Pavement formed of stone coated and cemented together with relatively pure Portland cement grout, the voids between the stones so coated being filled with bituminous cement.
- U. S., 1,003,693, Sept. 19. J. B. BESHORE, Harford Co., Md. Composition for making bricks, consisting of SiO<sub>2</sub> 58, FeO 11, Al<sub>2</sub>O<sub>3</sub> 22, MgO 1 and Na<sub>2</sub>O or K<sub>2</sub>O 2 parts.
- U. S., 1,003,730, Sept. 19. J. DE S. FREUND, Beaver, Pa. In molding cement and concrete, applying to the mold surfaces an adherent transferable coating containing soapstone and oil, and then shaping the material in the mold, thereby transferring a portion of the coating to the article to protect it.
- U. S., 1,003,813, Sept. 19. W. SCHUMACHER, Osnabrück, Ger. Increasing the binding power of basic calcium silicates, by the addition of fine quartz powder, the total amt. of silicate present in the mixt. thus formed not to exceed the amt. of lime, and also adding if desired other materials, such as lime, gypsum, asphaltum and stearin, and subjecting the entire mass to the action of steam under pressure.

Brit., 22,202, Sept. 24, 1910. J. T. HOLL, Prince of Wales Hotel, De Vere Gardens, Kensington, London, S. W. A cement or compd. consisting of a mixt. in suitable proportions of leather filings or granulated leather, plaster of Paris, powdered or ground gum, glue, or the like, and powdered or ground glass or similar material, said mixt. being worked into a paste or moist mass for use by the addition of a suitable liquid adhesive or H<sub>2</sub>O.

# 21. FUELS, GAS AND COKE.

#### I. D. PENNOCK.

Practical Notes on Coal. MICHABL DODD. J. Chem. Met. Min. S. Africa, 12, 14-5.—Reply to discussion (cf. C. A., 5, 1986). A general statement of the relative qualities of the Transvaal coals by districts is given. The calorific values and volatil contents, respectively, of the various districts are as follows: Springs, 10.3-5, 20-31; Vaal River and O. R. C., 10.3-5, about 27; Heidelberg, 11, 21; Middleburg, 12.5-13.1, 22-9; and Natal, 13.5-14, 19-26. In Natal small amts. of coke have been made, and a portion of the seam at Middleburg might be coked, but the Transvaal coals generally run too high in ash for such a purpose. The point is made that in changing

from one coal to another, few engineers pay attention to modifying the admission of air to their fuel, a matter which in some cases may be important.

E. Waller.

Anthracite Culm Briquets. C. DORRANCE, JR. Bull. Am. Inst. Mining Eng., 57, 693-718.—The writer reviews historically the manuf. of briquets from the waste of anthracite mines and gives an account of expts. of recent date on anthracite culm briquets.

C. A. COLE.

Diesel Oil Engin. C. Day. Engineering, 92, 369-70.—A discussion of the use of the Diesel engin as an economical generator of power as compared with other types of engins.

C. A. COLE.

Producing Power from the Sun's Rays. A. D. BLAKE. Power, 34, 506-7.—An illustrated description of the successful Shuman plant at Philadelphia. By a system of mirrors the rays are concd. on a steam boiler; the steam in turn is used to drive elec. generators.

L. K. MULLER.

Smoke Abatement. J. S. OWENS. Engineering, 92, 370-1.—O. discusses the present methods for detg. the vol. and d. of smoke emitted by chimneys and suggests some new factors and the use of a small telescope. The telescope has fixed in the optical system at a point which may be brought into focus of both object-glass and eye-piece, a revolving eccentric disk containing a number of graduated smoked glasses arranged in cells around the center.

C. A. COLE.

The First Natural Gas Pipe Line in the Galician Oil Regions. K. Feldman. Petroleum, 6, 2232-5.—The gas is colorless and has a slight odor of benzine. Two analyses of gases from different wells give 8.7% by vol., to 8.8% of heavy hydrocarbons  $(C_nH_{2n})$ ; light hydrocarbons  $(CH_4)$ , 86.5, 83.1; O, 1.0, 1.7; N, 3.8, 6.4; air content about 5.0, 8.0. The calorific value is 10.835, and the sp. gr., 0.750. Details of the construction of the pipe line from Tustanowice to Drohobycz and machinery are given. Since the gas possesses but little odor, it will be treated, as it enters the line, with mercaptan  $(C_2H_4SH)$ , so that leaks in the line may be easily detected.

O. E. Bransky.

Tarry Distillate in the Gas Works. H. LAURAIN AND SAINTE-CLAIRE DEVILLE. Rev. gén. chim., 14, 193-9.—Under ordinary conditions 1 cu. m. of gas as it issues from the main carries tar 200 g., ammoniacal water-vapor 200 g., CaHa vapor 42 g. and C<sub>10</sub>H<sub>2</sub> 17.5 g. On issuing at 65° all but 5% of the tarry vapor passes into the liquid state, but only 1/2 of this deposits in the main; about 90 g. per cu. m. is still carried on in the form of finely divided drops and 10 g. per cu. m. as uncondensed tar. The gas may be freed from this tar by clarifing while still hot and then cooling down, or by cooling before clarification. In the 1st case the suspended tar is caused to condense by "shocking" the gas by passing it slowly through a settling chamber without appreciable cooling. The only advantage is that the tar being removed at a high temp. the gas retains all the C.H. vapor. On the other hand the gas remains satd. with  $C_{10}H_{2}$  at a high temp, which on cooling deposits as free  $C_{10}H_{2}$ , there being no tar to act as a solvent. When cooling precedes clarification, the tar while still intimately mixed with the gas, dissolves all the C<sub>10</sub>H<sub>2</sub> and becomes satd. with C<sub>2</sub>H<sub>4</sub> by dissolving 2 g. out of the total of 42 g. of C.H. in a cu. m. of gas. When the clarification takes place in the cold the vapor tension of the C<sub>10</sub>H<sub>8</sub> dissolved in a large quantity of tar is far below that of pure  $C_{10}H_8$  at the same temp. The 2nd process is evidently the more efficient. The rapid cooling of the gas also insures complete removal of NH<sub>2</sub>. G. N. TERZIEV.

Porcelain Kilns Using Generator Gas (STAMPE). 19.
Liquid Fuel Effects Great Economy. 22.
Photometry of Lights of Different Color (PIRANI). 2.



Photometric Flame Standards (BUTTERFIELD, et al.). 4. Gas Compared with Electric Light (SCHLESINGER). 4. Constituents of Coal (PICTET, RAMSEYER). 10.

Carburetted Water Gas. Philadelphia: United Gas Improvement Company. 82 pp. Presentation Edition.

Illumination: Its Distribution and Measurement. New York: MacMillan. \$2.75.

KARPENNO: Die Entropietafeln für Gase und ihre Verwendung zur Berechnung der Verbrennungsmaschinen. Berlin: Boll & Pickardt. 1.50 M.

- U. S., 1,001,028, Aug. 22. N. H. HENDERSON, Vernon, N. Y. Purge-valve for gas-producer plants.
- U. S., 1,001,398, Aug. 22. J. A. HERRICK, New York, N. Y. Gas-producer with a casing including a fixed upper section and a revoluble lower section, the upper section having a depending annular curtain spaced from its walls and terminating near the junction of the sections, a blast being delivered within the annular curtain and also within the lower section and gases being discharged from the space between the annular curtain and the wall of the upper section.
- U. S., 1,001,509, Aug. 22. J. C. CROMWELL, Cleveland, Ohio. Stirrer for gas-producers.
- U. S., 1,001,543, Aug. 22. D. McDonald, Louisville, Ky. Coke-quenching apparatus.
- U. S., 1,001,738, Aug. 29. J. P. DOVEL, North Birmington, Ala. Gas-cleaning apparatus.
  - U. S., 1,001,739, Aug. 29. Idem. Gas washer, for blast furnace gases.
  - U. S., 1,001,740, Aug. 29. Idem. Gas-cleaning apparatus.
- U. S., 1,001,953, Aug. 29. J. A. HERRICK, New York, N. Y. Gas-producer with a casing having upper and lower sections, the lower section being revoluble, a depending revoluble curtain in the upper section to contain raw fuel and form an upper combustion chamber and mechanism for delivering a blast into the lower part of the casing and also within the curtain.
- U. S., 1,002,311, Sept. 5. S. M. QUINN, Iola, Kans. Gas-producer having an upright cylindrical body having a free lower edge, a rotary member provided with a water seal arranged to receive the lower edge of the body, and hollow grate arms in said member in open communication with the water seal and having a central discharge opening.
- U. S., 1,002,697, Sept. 5. G. H. ISLEY, Worcester, Mass. Gas producer having a rotating ash pan, a radial scraper in contact with the bottom and side of the pan and supported by the pan, the scraper being capable of rotation with the pan and having mechanism for locking it in stationary position during the rotation of the pan.
- U. S., 1,002,810, Sept. 5. N. LATTA, Milwaukee, Wisc. Assignor to Allis-Chalmers Co., same place. Gas washer.
- U. S., 1,003,147, Sept. 12. W. H. MURRAY, San Francisco, Cal. Composition for treating fuel (e. g., slack) to promote its combustion, consisting of mixed Na<sub>2</sub>SO<sub>4</sub> and Ca(OH)<sub>2</sub>, both in dry, ground condition, the proportion of Ca(OH)<sub>2</sub> being at least <sup>1</sup>/<sub>8</sub> that of the Na<sub>2</sub>SO<sub>4</sub>. About 3% of the mixt. is sprinkled over the fuel.
- U. S., 1,003,296, Sept. 12. W. D'A. RYAN, Watertown, S. D. Making hriquets for fuel, by comminuting straw, adding a surplus of binder, e. g., coal tar or molasses,

thereto sufficient to moisten the interior surfaces of the straw, removing the excess of binder by press rolls, and compressing the remaining mixt, in the form of a sheet.

- U. S., 1,003,524, Sept. 19. S. B. Shilldon, South Bethlehem, Pa. Gas producer structure.
- U. S., 1,003,544, Sept. 19. L. L. SUMMERS, Chicago, Ill. Coking furnace with a number of retorts, a conveyor floor in the retorts and mechanism for moving the floor through the retorts and from one retort to the other in a continuous path.
- U. S., 1,003,559, Sept. 19. M. C. WHITAKER and F. J. METZGER, Yonkers, N. Y. Storage receptacle for acetylene, formed wholly or partially of brass or Cu and containing a porous filler, e. g., asbestos, acetone, and NH<sub>4</sub>CNS to prevent the formation of metallic acetylides.
- U. S., 1,003,887, Sept. 19. A. B. Duff, Pittsburgh, Pa. Apparatus for recovering ammonia from gas-producer gas, comprizing in sequence a gas producer, a conc. device for b. down the weak soln. of  $(NH_4)_2SO_4$ , a heat interchanger in which  $H_2O$  is heated to or near the b. p., and an  $NH_2$  recovery tower in which the cooled gas is treated as usual, together with a second heat interchanger above the first, and connections for leading the heated  $H_2O$  from the lower to the top of the upper heat interchanger and for leading up through the latter a counter-current of air so that a mixt. of steam and air is produced and for leading the cooled  $H_2O$  from the upper to the lower heat interchanger to wash the gas so that the circulation is continuous, and for leading the mixt. of air and steam from the upper heat interchanger to the gas producer.
- U. S., 1,003,969, Sept. 19. W. J. Paul, J. H. Lynce and J. G. Meyer, Matteawan, N. Y. Method of perfecting combustion by effecting primary combustion, combining O with the unconsumed gases resulting from the primary combustion, sepg. the combined gases into distinct bodies, causing them to flow through a zone of secondary combustion and continuing them therein long enough to produce substantially complete combustion, radiating a portion of the heat from the secondary combustion to the body to be heated and applying a portion of the heat thus generated to the sepd. bodies of gases to promote and continue combustion thereof.
- Brit., 16,981, July 16, 1910. W. DIXON, 30 Kelvinside Gardens, Kelvinside. An app. for treating bituminous coal, in which carbonaceous material is treated step by step, of the type having an upper part which is heated, an intermediate part which is cooled, and a lower part which is curved and H<sub>2</sub>O sealed, and comprizing a retort having 3 portions the relative capacities of which are such that the quantity of material which after carbonization fills the upper part is, when such material has descended into the intermediate and lower parts, resp., just sufficient to fill the same, the retort being formed with a tapered upper portion with its larger end downwards, the intermediate portion being parallel and the lower portion extended and curved.

Brit., 20,875, Sept. 7, 1910. E. HENSS, Soden, Taunus, Hasselstrasse, Ger. Obtaining ammonium sulfate from gases of dry distillation by depriving the gases of tar in a hot state and drawing them off by an acid jet app.

# 22. PETROLEUM, ASPHALT, COAL TAR AND WOOD PRODUCTS.

R. E. HUMPHREYS.

History of Petroleum. XII. C. v. KLINCKOWSTROEM. Petroleum, 6, 2244.

O. E. BRANSKY.

The Estimation of Aromatic Hydrocarbons in Petroleum. E. SEVERIN. Men.

du. petrole Roum., 1911, 21, 22; through Petroleum, 6, 2245-6.—The Nastiukov formolite reaction is uncertain, since the amt. and comp. of the ppt. is variable. The pure paraffins (normal C<sub>2</sub>H<sub>14</sub> and C<sub>2</sub>H<sub>18</sub>) nor the satd. naphthenes (Me-cyclohexane, di-Me-cyclohexane) are not attacked by the CH<sub>2</sub>O and H<sub>2</sub>SO<sub>4</sub> mixt.; the olefines (amylene, octylene) yield a reddish brown syrupy liquid; the unsatd. naphthenes (cyclohexene, p-Me-cyclohexene and pinene) give a reddish brown ppt. easily sol. in H<sub>2</sub>O; the aromatic hydrocarbons give a red ppt., insol. in H<sub>2</sub>O, with the exception of naphthalene, which yields a deep green ppt., and anthracene which gives a yellowish green ppt. Even traces of aromatic hydrocarbons and unsatd, naphthenes give a characteristic red coloration. S. suggests the use of this reaction for detg. the purity of the paraffins prepared by Sabatier's method. In the case of pure hydrocarbons, it was found that a definit amt. of H.SO, always reacted with a definit amt. of the hydrocarbon. In detg. the amt. of aromatic hydrocarbons S. treats the fractions below 200° with 3% conc. H<sub>2</sub>SO<sub>4</sub> to remove the olefines and then ppts. with the CH<sub>4</sub>O-H<sub>2</sub>SO<sub>4</sub> mixt. Where large amts, of aromatic hydrocarbons are present, the sample should be dild. with paraffins upon which blanks have been previously made with the reacting mixt. In the fractions above 200°, treatment with 3% conc. H<sub>2</sub>SO<sub>4</sub> is not sufficient to remove all of the olefines. S. resorts to bromination. He has succeeded in limiting the action of Br to the olefines O. E. BRANSKY. and unsatd. naphthenes only.

An Observation upon Petroleum. J. WALTER. Chem. Tech. Zig., 29, 129-31. -In cleaning with conc. H<sub>2</sub>SO<sub>4</sub> and HNO<sub>5</sub> the vessels in which attempts had been made to purify a by-product in the manuf. of aniline dyes with a petroleum product, a marked odor of musk was detected. The substance causing this odor was shown to be present in the oil by treating the latter with conc. H.SO, or with fuming acid (containing 20% SO<sub>2</sub>), warming the acid after sepn. from the oil with HNO<sub>2</sub>, and pouring the mixt. into water, when the odor of musk was noticed. In the partial combustion of petroleum distillates, W. observed that, although the original oil did not give the odor, its products gave a marked odor. The oil employed was an American light distillate. It is possible that the manuf. of artificial musk from the sludge acid could be profitably undertaken, and the origin of a petroleum might be detd. by this odor test. The odor is caused by trinitrobutyltoluene. W. describes the prep. of organic substances from waste material in the presence of asbestos coated with Cu and V. The petroleum distillate, b. above 300°, is sprayed into a current of air mixed with steam; the mixt. is conducted over glowing asbestos covered with fine CuO, and the gases condensed. The distillate is strongly acid, and the bottom layer of H<sub>2</sub>O contains AcOH and its homologs. An organic acid, solid at oo, is pptd. by a mineral acid from the soda soln. of the upper oil layer. An aldehyde, peppermint odor, is pptd. with a NaHSO, soln. An oily liquid, half of which distils between 140° and 300°, is the main product of the combustion. This process affords a means of cracking petroleum distillates. The product b. between 140-300° gave the musk odor.

O. E. BRANSKY.

Analysis of Ceresin and Paraffin Mixtures. N. CHERCHEFFSKY. Mat. grasses, 4, 37; through Petroleum, 6, 2128.—Since ceresin and paraffin closely resemble each other chemically, physical tests must be applied to distinguish them. In the analysis of com. ceresin, the saponifiable constituents must be sepd. from the unsaponifiable by the ordinary methods of fat analysis, so that all tests may be applied in a fraction containing only ceresin and paraffin. Solubility, critical soln. temp., turbidity pt., and n are the chief distinguishing marks. The solvents used for the solubility test are CS<sub>2</sub> and CCl<sub>4</sub>. The best solvent for detg. the critical soln. temp. is 96.5% MeOH. The ceresin content of a mixt. is calc. from the equation, C = 100(T - t')/t - t',

in which C is % ceresin, T the critical soln. temp. of the mixt., t the critical soln. temp. of ceresin, and t' that of paraffin. The solvent used in detg. the turbidity pt. is  $C_*H_*$ . Since the turbidity pt. of the mixt. is the arithmetical mean of the turbidity pts. of its components, the % content of the latter can be easily calc. The n of ceresin and paraffin differ enough so that by interpolation the quant. comp. of the mixt. can be detd.

O. E. Beansky.

The Origin of Oil and Gas. Anon. Petroleum Rev., 24, 303-4, 335.

R. E. HUMPHREYS.

Liquid Fuel Effects Great Economy. Anon. Petroleum Rev., 24, 293.—A trial of oil fuel at 6 c. per gallon on the revenue cutter "Golden Gate" for 3 months amtd. to \$153.32 and of coal at \$5.40 per ton \$694.44. The cost per mile for oil was 6.9 c., and 54 c. for coal. The personnel was reduced from 4 to 3 men. R. E. H.

Petroleum in North Staffordshire. Petroleum Rev., 24, 301.—An historical account of the methods used in the production and refining of the crude oil. R. E. H.

The Chemical Composition of Naphtha Gas. I. GRUSZKIEWICZ AND I. HAUSMAN. Ropa, 1911, 7; through Petroleum, 6, 2245.—Six analyses of Galician natural gas show that it contains neither CO, nor CO, differing in this respect from the gases of the Roumanian provinces. Besides air, the gas contains only pure hydrocarbons. Its calorific value is 10,835. It is especially suitable for gas motors.

O. B. Bransky.

Natural Asphalt, Asphaltite, Petroleum, and Natural Gas in Okiahoma. HUTCHIN-SON. Oklahoma Geo. Surv. Bull. 2, 1911; through Petroleum, 6, 2127.—The geological section of this report contains the following classification of the natural hydrocarbons and related substances, as well as the natural and artificial bitumen compds. Natural hydrocarbons: I. Gaseous; (a) well gas, (b) natural gas. II. Bituminous; A. liquid; (a) naphtha, (b) petroleum; B. viscous; (a) maltha, (b) mineral tar, (c) brea, (d) chapapote; C. elastic; (a) elaterite, (b) wurtzilite; D. solid; 1. asphaltite; (a) albertite, (b) imphonite (c) grahamite, (d) nigrite, (e) gilsonite; 2. coals; (a) lignite, (b) sub-bituminous, (c) bituminous, (d) semi-bituminous, (e) semi-anthracite, (f) anthracite; E. resinous; 1. amber; (a) ketitnite, (b) gedirnite, (c) glossite, (d) kumanite, (e) simetite, (f) krantzite, (g) etemawinite; 2. copalite; 3. bathoillite; 4. geocerite; 5. leucopetrite; Wax-like; 1. ozocerite; (a) zietritikite; (b) chrismatite, (c) urpethite; 2. hatepetite; 3. nopalite. V. Cryst.; 1. ficktelite; 2. paraffin; 3. scheerasite; 4. gartite; 5. adriabite. Natural and artificial bitumens: I. Natural; A. As impregnation; 1. asphaltic limestone; 2. sand or sandstone; 3. slate; 4. clay; B. liquid; 1. refinery residues; 2. sweating of petroliferous rocks; II. Artificial; A. viscous; 1. gas tar; 2. pitch; B. solid; 1. refined Trinidad asphalt; 2. mastix or asphaltite; 3. asphaltmastix (gritled); 4. plastering material; 5. roofing material. The history, chemistry, origin, and production and occurrence of oil in Oklahama is described and discussed. O. E. Bransky.

Injurious Effect of Alcohol Denatured with Wood Spirit on Worker's Health. HORBACZEWSKI. Oesterr. Chem. Ztg., 14, 139-41; through J. Soc. Chem. Ind., 30, 826.—The use of wood spirit should no longer be permitted for denaturing EtOH because the health of those working with such denatured EtOH is liable to be seriously affected by the impurities contained in the wood spirit together with the greater toxicity of the MeOH itself in comparison with EtOH. The use of shellac is advocated as a denaturing agent for alc. for polishing and lacquering industries. R. E. H.

Methyl Alcohol in the United States. Chem. and Druggist, Aug. 26, 1911; through J. Soc. Chem. Ind., 30, 1106.—The New York State Pharm. Assoc. and the Nat. Assoc. of Retail Druggists believe that a national law ought to be enacted, such as exists in Minnesota, requiring that the name "wood naphtha" be used for "wood alc." to avoid its being confused with EtOH.

R. E. H.

Tests for Asphalt Cements (HOWARD). 20.

DELEHAYE, H.: Huiles minérales. Paris: C. Béranger. 215 pp., 4 Fr.

ENGLER, C.: Ueber Zerfallsprozesse in der Natur. Leipzig: S. Hirzel. 1.25 M.

ENGLER, C. AND HÖFER, H.: Das Erdől, seine Physik, Chemie, Geologie, Technologie und sein Wirtschaftsbetrieb. (5 Bände.) Band III. Technologie des Erdőls und seiner Produkte. Leipzig: 8°, 56 M.

- U. S., 1,001,937, Aug. 29. C. ELLIS, Larchmont, N. Y. In making oxalates, passing a continuously moving stream of material containing alkali and woody fiber through a heated zone maintained at oxalate-forming temp., and shielding the material from free access of atm. air while impinging regulated jets of air upon portions of the stream.
- U. S., 1,002,034, Aug. 29. H. O. CHUTE, Cleveland, Ohio and K. P. McElroy, Washington, D. C. In making acetates, passing a granulated mass containing woody fibrous material, previously treated with an alkali soln., such as waste soda pulp lye, past a current of hot dil. air, traveling in the opposit direction, the temp. of the air and its diln. being regulated to cause formation of acetates, carbonizing by a higher temp. (about 400°) out of contact with air, and sepg. the carbonate and acetate from the carbonized mass.
- U. S., 1,002,474, Sept. 5. M. J. TRUMBLE, Los Angeles, Cal. Oil-refining apparatus comprizing a fire-still, a vapor outlet pipe therefrom, a H<sub>2</sub>O jacket around the pipe, a sepg. app. having a series of sepg. compartments, the first of which is connected to said outlet pipe, an overflow connection being provided between each compartment and the succeeding compartment, an oil outlet from the last compartment, a condensing chamber extending over all of the compartments and communicating with their upper portions, a supply for cooling fluid for the condenser and for steam to the lower portions of the compartments, and a valved by-pass from the inlet to the outlet of the sepg. app.
- U. S., 1,002,570, Sept. 5. J. A. Dubbs, Santa Monica, Cal. Treating petroleum, to free it from H<sub>2</sub>O, distil or produce asphalt from it, by continuously causing the petroleum to drop, in vacuo, in the presence of ascending currents of combustion gases to drive off the lighter products from each of the drops while in suspension and collecting the asphaltic residuum below the heat zone, the size of the drops being varied according to the temp. of the ascending gases and the product desired.
- U. S., 1,003,040, Sept. 12. F. H. DUNHAM, Los Angeles, Cal. Obtaining asphalt from asphaltic oils by heating the oil to 205-315° within a shell, removing oil from the lower portion of the shell to the upper portion and there maintaining a charge of the removed oil, subjecting the latter to the temp. of the shell above the surface of the lower charge of oil, causing a flow of oil from the upper to the lower charge and removing the heavier vapors and condensate from the shell below the level of the top of the upper charge.

#### 23. CELLULOSE AND PAPER.

#### A. D. LITTLE.

New Researches on Cellulose, 1910. II. W. VIEWEG. Wochbl. Papierjabr., 42, 3541-5.

Industry of Plaster Substances: Cellulose Acetate. L. CLEMENT AND C. RIVIÈRE.

Rev. chim. ind., 22, 215-20.—The article reviews some of the methods for the prep, soln. and applications of cellulose acetate. It includes some new information on relative saponification values of different acetates, and a stretch curve of a semi-elastic plastic cellulose acetate compd.

V. Nunez.

Cellulose Acetate. L. CLÉMENT AND C. RIVIÈRE. Caoutchouc and gutta-percha, 8, 5449-50.—An argumentive article, comparing the uses of cellulose acetate and cellulose nitrate. The acetate has practically displaced the nitrate for the manuf. of cinematograph films and in addition may be used to make an excellent grade of artificial silk. For molded articles, the nitrate may be said to have the advantage on account of lower cost.

V. Nunez.

Extracts from the Literature of Cellulose and Paper Chemistry. C. G. SCHWALBE. Darmstadt. Papier Ztg., 36, 2496-7, 2531, 2667-8, 2699, 2735-6, 2769-70. V. N.

The Sizing of Paper. C. G. Schwalbe. Papier Ztg., 36, 2768-9.—A review of recent advances in this field.

V. N.

Use of Magnesium Sulfate for Rosin Sizing. PAULI. Wochbl. Papierjabr., 42, 2791-2.—A series of practical tests were made to det. whether kieserite might replace alum in rosin size. Pptn. of the rosin with MgSO<sub>4</sub> alone produced no sizing effect, while mixts. of ½ kiersite and ½ alum produced moderate sizing. This, in some measure, contradicts the work of Frohberg, who found that mixts. of Mg and Al sulfates produced about the same sizing effect as MgSO<sub>4</sub> alone.

V. Nunez.

The Reaction between Pigment, Loading Material, and Sizing in Paper Pulp. L. KOLLMANN. Papierfabr., 9, 845-53.—The sol. dyes used in coloring paper are capable of reacting with the loading material and the sizing agent as well as with the fiber itself. Basic dyes show the greatest affinity for the acid loading materials. e. g., kaolin. They give a colored ppt, with free rosin in the sizing. Acid dyes color the basic loading agents, e. g., magnesia, most strongly and react with size-alum ppt. but not with free rosin. Substantive dyes are absorbed more readily by basic than by acid loading.

V. Nunez.

Manufacture of Parchment Papers. Anon. Wockbl. Papierjabr., 42, 3055-7.

—A discussion of the kind of stock which should be used for making parchment paper, Rittner-Kellner cellulose and Mitscherlich cellulose being variously advized. V. N.

The Microscopy of some Paper Pulps. T. F. HANAUSEK. Papierfabr., 9, 731, 754-5.—A microscopic study of Trewia, Bombax, and of Bhabur-grass cellulose, cellulose of Pinus excelsa Wall and of Pinus longifolia (three-needle pine), with illustrations.

V. NUNEZ.

Detection of Wood Pulp Liquor in Tanning Extracts. Anon. World's Paper Trade Rev., 56, 390.—Waste sulfite liquors by suitable treatment yield a tanning ext. much resembling oakwood ext. When used alone this produces a very inferior leather, but it may be used to adulterate other exts. without causing any decided differences in the appearance of the leather. Procter and Hirst offer the following method of identification of waste sulfite liquor in tanning exts. When 0.5 cc. of aniline is added to 5 cc. of tanning ext., a turbid emulsion is produced. In the case of unadulterated exts., the turbidity disappears on addition of 2 cc. conc. HCl. If wood pulp liquor be present a decided ppt. remains.

The Bleaching of Wood Pulp. IV. R. W. SINDALL AND W. BACON. World's Paper Trade Rev., 56, 385-6.—The color readings obtained for a series of bleached pulp in which the % of bleach used ranged from 2-24% are given by means of tables and curves. Results are given for both hot and cold bleached pulps. The amt. of coloring matter as measured by a tintometer decreases uniformly as the % of bleaching powder rizes.

V. Nunez.

Mode of Working up Bought Kraft Pulp. Anon. Papierjabr., 9, 1001-2.—A technical description.

V. N.

Deterioration of Colored Papers. H. A. Maddox. World's Paper Trade Rev., 56, 392.—The main factors leading to the deterioration of colored papers are as follows: (1) Character of the fibrous material in the paper. Rag stock may be considered as permanent while wood, straw, and esparto fibers deteriorate with more or less rapidity. (2) Quality and permanence of the dye. Coal tar dyes are chiefly used in the usual grades of colored printing and writing papers and are subject to change unless the papers are carefully stored. (3) Action of sunlight, moisture, and air. In combination these cause fading and in case of wood pulp discoloration to a dirty brown, the latter being due to an attack on the fiber itself. If the paper is rosin sized, an excess of free rosin is changed chemically by sunlight and discoloration results. It is imperative that colored paper, especially the cheaper grades, should be stored in the absence of sunlight and moisture, although moisture and air, unaided by sunlight, do not cause rapid deterioration in the color.

Raw Materials for Paper from Kamerun. Anon. Papierjabr., 9, 914-7.—Cotton-wood pulp contains fiber of good av. length (1.9 mm. by the sulfite process) but is difficult to bleach owing to a sol. brown substance which is found in the medullary rays. A yield of 42.7% unbleached pulp was obtained by the soda process, the wood being boiled with 4% NaOH soln. for 3½ hrs. at 8 atmospheres pressure. This pulp could be bleached with 24% bleaching powder. The sulfate process gave a yield of 43.8% unbleached cellulose. The sulfate process yielded an unbleachable pulp. Ibid., 948-50.—Elk wood pulp is very similar to that made from cotton wood, but the fibers are shorter and thicker. The yields were slightly higher. The pulp is even more difficult to bleach. Tests of paper made from elk wood and cotton wood show that the fibers are strong and well capable of felting, but bleaching greatly decreases their strength.

V. Nunez.

Music Paper. Anon. Papierjabr., 9, 877-8.—The paper employed for edition of musical works is usually made from cotton and linen rag stock in various admixtures with esparto, pine, straw, and old paper pulps. The sizing is resin glue with some starch. But little loading is used. The pulp is beaten with semi-sharp bars and worked in the machine at slow speed.

V. Nunez.

Paper as a Substitute for Gold-beater's Skin. W. Theobald. Papierjabr., 9, 979-82.—No paper has yet been made which is entirely satisfactory for this purpose, although a thin paper, soaked in borax or alum soln. and coated first with albumin and then with a soln. of isingless has been used to some extent.

V. Nunez.

Hubbard, E.: Die Verwertung der Holzabfülle. 3 Aufl. Wien: 8°, 216 pp., 3 M.

MÜLLER, M.: Literatur der Sulfitablauge. Berlin: 8°, 117 pp., 6 M.

U. S., 1,001,446, Aug. 22. S. R. RIEGLER, Budapest, Austria-Hungary. Making a plastic material from waste paper by softening 1 kg. of the paper in cold H<sub>2</sub>O, b. the paper in H<sub>2</sub>O, disintegrating the mass and pressing it to remove H<sub>2</sub>O, again disintegrating and adding 30 kg. of dil. glue soln., 0.25 kg. dil. starch soln., 10 kg. water glass, ZnCl<sub>2</sub> soln. and ZnO, kneading and drying the mass.

U. S., 1,001,521, Aug. 22. O. F. HAKANSSON, Nyqvarn, Sweden. Paper-making machine.

U. S., 1,001,822, Aug. 29. W. CURTIS, JR., Palmer, N. Y. Supplying stock to

paper-making machines so as to maintain a uniform distribution of stock in the stuffchests of several machines.

- U. S., 1,002,017, Aug. 29. T. E. WARREN, Ticonderoga, N. Y. Vacuum thick-ening and washing machine for paper-stock.
- U. S., 1,002,634, Sept. 5. E. Brandenberger, Thaon-les-Vosges, France. Apparatus for drying films of cellulose such as obtained from an aq. soin. of cellulose xanthate.
- U. S., 1,003,606, Sept. 19. T. J. HUTCHINSON, Bury, England. Apparatus for washing fibrous paper-making material from bagasse.

# 24. EXPLOSIVES.

#### C. E. MUNROE.

A Remedy for Blasting Fumes. R. J. Lewis. Arms and Explosives, 19, 62.—Expts. with "Oxozone," a material for the neutralization of deleterious fumes due to blasting (Wm. Burgess, Br. pat. 12,988, 1909). Its comp. is 24 parts KNO<sub>2</sub>, 3 blasting powder, 2CuO<sub>2</sub>, 1 flour or C. It is prepared in wrappers containing about 3 oz., or unwrapped for charging directly with the explosive. Mine air samples taken at the face before firing showed no CO. 10 min. after firing 29 plugs of gelignite ( $\frac{1}{2}$ ), 15 ft. fuse, no "Oxozone," — CO = 0.067%. 7 min. after firing 27 plugs gelignite, 15 ft. fuse, 15 oz. "Oxozone," — CO = nil. A number of results of tests are shown in a table.

C. G. Storm.

The Cause of Gun Erosion and its Relation to the Use of Nitro-powders. P. Rusch. Z. ges. Schiess-Sprengstoffw., 6, 253; from Mitt. Seewesen.—Erosion can never be entirely prevented but may be appreciably reduced by the use of a suitable explosive. The chief causes of erosion are: (1) The chem. action of gases, e. g., oxides of N, on the steel; (2) the action of solid particles in the products of explosion of the powder; (3) the high temp. of explosion. The third point is of the greatest importance. At temp. of 200° the strength of steel increases, but beyond this temp. it falls rapidly. At 500° the tensil strength of cast steel is only about ½ its value at 20°. Under conditions of rapid firing, erosion is more than double that under ordinary firing, because of the high temp. reached by the metal. There appears to be a critical temp. beyond which erosion increases rapidly with higher temp. and pressure. The duration of action of the hot gases is also considered; thus, long gun barrels and thick-walled powder grains tend to cause greater erosion. A nitrocellulose powder of about 12% N gives a much lower temp, than higher nitrated powders, with consequently less erosion. C. G. STORM.

The Measurement of Time in Ballistics. KARL v. BALTZ. Z. ges. Schiess-Sprengstoffw., 6, 330-2.—B. describes the principle of the Boulangé chronograph, the Wheatstone chronoscope, the Smith chronograph, and photographic methods for detg. the velocity of shells.

C. G. STORM.

The Cost of Electric Ignition Compared with that of Fuse Ignition. E. Bulgis. Z. ges. Schiess-Sprengstoffw., 6, 352; from Ker Bergbau, 23, 314, 341.—Practical comparison in a coal mine where an av. of 12,000 caps was used monthly, showed a reduced cost per ton by elec. ignition. The cost per shot being materially higher, a decrease in number of shots with this method of firing is evident.

C. G. Storm.

The Explosion on the French Battleship "La Liberte." EDIT. Engineering, 92, 432, 463-4.—The explosion completely wrecked the vessel, tossing a piece of armor plate weighing 37 tons upon the "Republique" lying at a distance. It is alleged that 2 re-

ports were heard and that 19 minutes later the vessel blew up. This lapse of time is stated to agree with the occurrence on the "Jena" (C. A., 1, 1323). Both explosions are attributed to spontaneous combustion of smokeless powder, and this time interval conforms "to what might be expected to happen under certain conditions with this powder." The article is illustrated with 3 striking cuts. Charles E. Munror.

### Manufacture of Nitrogen Compounds (Scott). 4.

- U. S., 1,001,072, Aug. 22. E. I. DU PONT, Wilmington, Del. Apparatus for solidifying crystallizable mixtures (e. g., black gunpowder mixt.) without substantial crystallization and forming strips thereof.
- U. S., 1,001,992, Aug. 29. H. SANBORN, San Francisco, Cal. Cartridge for preventing noxious fumes being given off by explosives (such as nitroglycerin), containing (NH<sub>4</sub>)<sub>2</sub>CO<sub>2</sub> 2 and Ca(OCl)<sub>2</sub> 1 part.
- U. S., 1,002,824, Sept. 12. I. J. Cox, Du Pont, Wash. Assignor to E. I. du Pont de Nemours Powder Co., Wilmingtom, Del. Making oleum by causing a flowing body of H<sub>2</sub>SO<sub>4</sub> at an initial temp. between 35° and 46° to absorb SO<sub>5</sub> of a temp. within the same range, maintaining the ratio of the amt. of absorption acid to the amt. of SO<sub>5</sub> in the gas acted upon at not less than 43: 1 by wt., cooling the conc. acid to a temp. within the same range, and repeating the cycle until the desired degree of conc. is reached.
- U. S., 1,003,243, Sept. 12. A. ELLIS, Philadelphia, Pa. Assignor 11-12 to Kelly Mfg. Co., of Del. Apparatus for nitrating starch, comprizing a nitrating tank, receptacles for nitrating acids and material to be nitrated with connections therefrom to the nitrating tank and cooling coils around them, and a blower for forcing the finely divided starch through the supply connection into the closed nitrating tank, the latter having a hood to direct the particles downward into the acid.

### 25. DYES AND TEXTIL CHEMISTRY.

### L. A. OLNEY.

Cotton Wax. Edmund Knecht and John Allan. J. Soc. Dyers Colourists, 27, 142-6.—Cotton wax was first isolated by Schunk, 1868, who obtained 0.004%. With modern methods the authors have repeated the extraction of cotton, using a Soxhlet app., first with petroleum ether, and then with benzole, and the wax which it contained was obtained in 2 fractions. The 1st product was an odorless, dull yellow substance, closely resembling beeswax in tecture and fracture. When repeatedly treated with b. 96% alc., 18.82% of an insol., glassy wax was left, m. 78°, I.no. 11.28. The portion sol. in alc. was more plastic, m. 62°, I no. 33.42. When boiled with 0.5 N glycerol NaOH soln., it was found to contain 47.5% unsaponifiable matter, m. 67-8°. Upon acetylation 2 hydrocarbons of cryst. form were obtained, one m. 68.1°, and giving on analysis results indicating hentricontane (CaHe), and the other m. 70° and agreeing in analysis with dotriacontane. From the acetylated alc. a crop of crystals sepd. on cooling its alc. soln. They melted at 127.2°, and the substance was evidently a phytosterol. By sepg. and acidifying the saponified portion of the wax 0.21% of glycerol was isolated, a small quantity of an acid m. 74.4°, and stearic and palmitic acids. The fraction of wax obtained from benzole was a dark green granular mass m, 68°, acid no. 4.03, sapon. no. 83.3. The unsaponified matter yielded 33.55% of a reddish brown sticky wax, m. 63.4°. Upon acetylation a considerable quantity

of a phytosterol acetate, m. 122.4°, was obtained. From the fatty acids obtained from the saponified portion was sepd. a small quantity of cryst. acid m. 70.8°, and having a mean mol. wt. of 455. This is possibly melissic acid (C<sub>20</sub>H<sub>20</sub>O<sub>3</sub>). L. A. OLNEY.

Vat Dyes of the Anthracene Series. I. R. STAEBLE. Chem. Zig., 34, 731.—
A review of the anthracene and indigo vat dyes and their combination. A complete list of the most important groups and their derivs.

SIDNEY BORN.

Chemical Technology of Aniline Black A. G. Green. Mon. Sci., [4] 24, 235.—
The prep. and use of various aniline blacks, is described, and a summary of some of the work on the constitutional formulas given.

Sidney Born.

Review of the Brands of Dyes Recently Introduced into Commerce. F. REVERDIN. Mon. Sci., [5] 1, II, 642-55. J. J. M.

Theory of Color Processes. I. Coloring "Glanzstoff" with Crystal Violet. W. J. Müller. Chem. Zig., 34, 805-6; through Chem. Zentr., 1910, II, 768.—The absorption of crystal violet by "Glanzstoff" and cotton is expressed by  $X/m = 2C_{0.7}$  and  $X/m = 1.1C_{0.5}$ , where X = amt. of dye, m = amt. of material to be dyed, and C the final conc. of the bath. The exponent and const. are higher for "Glanzstoff" than for cotton, that is, cotton absorbs more dye for a dil. soln. than "Glanzstoff." Small amts. of oxycellulose and Ca(OCl)<sub>2</sub> remarkably increase the amt. of dye taken up by "Glanzstoff." "Glanzstoff" pptd. by NaOH is more changed by oxidation than that pptd. by acids. The fact that chem. changes of the fiber alter the behavior of it to dye is contrary to the observations of Freundlich and Losev (C. A., 1, 2343). The titration of crystal violet with TlCl<sub>2</sub> does not give const. results, but with certain precautions can be used. Dil. solns. of crystal violet use up more than the theoretical amt. of TlCl<sub>2</sub>.

Dyeing Wool with "Ciba" Blue, "Ciba" Red and "Ciba" Violet. S. KAPFF. Farber Ztg., 20, 105-7; through Chem. Zentr., 1909, I, 1618.—To det. how wool dyed with vat dyes compared with wool dyed with chrome dyes, several batches were dyed once with "Ciba" vat dyes and again with anthracene chrome black. These samples, as well as several undyed ones, were made into cloth. Results showed that the treatment with  $K_a$ CrO4 strongly affects the wool, while these samples dyed in the "Ciba" dyes in hydrosulfite-soda bath only lost 10% of their strength.

Spectroscopic Detection of Simple Lake Dyes on the Fiber. E. Grandmougin. Chem. Zig., 34, 1076-77.—For the spectroscopic detn. of naphtholazo dyes H<sub>2</sub>SO<sub>4</sub> soln. was found best, since the absorption spectra in xylene or glacial AcOH are less sharp; for the indigo dyes xylene is best; for the anthraquinone vat dyes conc. H<sub>2</sub>SO<sub>4</sub> or H<sub>2</sub>SO<sub>4</sub> + H<sub>2</sub>BO<sub>3</sub>. A table of the wave lengths for 28 of the most important dye of these 3 groups is given.

A Natural Substance Dyestuff. A. G. PEREIN. J. Chem. Soc., 97, 220; Proc. Chem. Soc., 26, 23.—The material known as the "red dura" of the Soudan consists of the leaf sheaths and stem of a variety of Andropogon sorghum or "Great Millet." The material was extracted in a Soxhlet extractor with acetone and yielded 14.7—16.27% of a dark brittle resin. It was purified by repeated soln. in cold acetone. The amorphous, greenish substance thus obtained seemed to be a Ca salt of the coloring matter. This crude material was fractionally crystallized from benzene repeatedly. In dyeing property, it closely resembles sanderswood (Ptorocarpus santalinus) giving with unmordanted wool a dull red shade, but this is rendered more permanent with a Cr or Cu mordant. The coloring matter, C<sub>16</sub>H<sub>12</sub>O<sub>4</sub>, called durasantalin, forms a scarlet cryst. powder sol. in alkalis with a violet red color. In fusion with alkali it yields phloroglucinol and p-hydroxybenzoic acid.

Indirubin. I. W. P. BLOXAM AND A. G. PERKIN. J. Chem. Sec., 97, 1460;

Proc. Chem. Soc., 26, 168.—In order to study the conditions necessary for the formation of indirubin by the air-oxidation of indoxyl, the authors have devized a method for the quant. est. of the former in indigo. The dye mixed with purified sand is extracted in a Soxhlet extractor with pyridine. The pyridine ext. is evapd. finally with the addition of hot water, and the ppt. which consists of indirubin together with a little indigotin and impurities, is collected on asbestos, washed with dil. HCl to remove mineral matter, with 1% alkali to remove indigo brown, and with 1% AcOH. The cryst. residue is dried, sulfonated, and the soln. of the mixed sulfonic acids analyzed by means of the Duboscq colorimeter. The residual mixt. of sand and crude indigotin is washed with hot 10% H<sub>2</sub>SO<sub>4</sub>, which removes indigo gluten and mineral matter, and with H<sub>2</sub>O until free from acid. The residue is then dried, sulfonated, the product dissolved in H<sub>2</sub>O, filtered and titrated with KMnO<sub>4</sub> or TiCl<sub>8</sub>. A quant. detn. of indigo brown is also possible by this method.

Formation of Isatin in Dyeing of Indigo. R. E. Crowther. J. Soc. Dyers Colourists, 27, 146-8.—Indigotin is lost during the dyeing process in the indigo vat. This loss is variously estd. from 5 to 30%. C. discusses various reactions taking place in the vat, but more particularly the formation of isatin. When a normal indigo hydrosulfite vat is oxidized, and the oxidized indigo filtered off, a yellow filtrate results, which on evapn. leaves a residue containing isatin and a S compd. which strongly resembles it; possibly it is the thio compd. corresponding to the pseudo isomeride of isatin. The presence of indoxyl having been detected in appreciable quantity in a normal hydrosulfite vat, C. suggests that the isatin is formed by the oxidation of indoxyl by H<sub>2</sub>O<sub>2</sub>, which is always present in an oxidized indigo vat. L. A. Olney.

Action of Sodium Sulfite and Bisulfite on some Azo Dyes. R. LEPETIT AND E. Levi. Lab. chim. Milano Ditta Lepetit, Dollfus and Gansser. Gazz. chim. ital., 41, I. 675-88.—From p-nitrobenzeneazosalicylic acid and Na<sub>2</sub>SO<sub>2</sub>, b. 3 hrs. in H<sub>2</sub>O, are obtained p-O,NC,H,NH,, H,NC,H,N,C,H,(OH)CO,H and an acid H,NC,H,(OH-(CO<sub>4</sub>H)SO<sub>3</sub>H, identical with Fischer's (Ber., 32, 81). With NaHSO<sub>3</sub>, the reaction is more energetic; at ordinary pressure, p-O<sub>2</sub>NC<sub>2</sub>H<sub>4</sub>NH<sub>2</sub> and H<sub>2</sub>NC<sub>2</sub>H<sub>4</sub>(OH)(CO<sub>2</sub>H)SO<sub>2</sub>H are formed directly (no intermediate formation of the aminoazo acid) and the mother liquors evolve NH<sub>2</sub> with alkalis and become blue and fluorescent. If the reaction is carried out in an autoclave, the aminosulfonic acid is still obtained, but there is no O.NC.H.NH. and the mother liquors yield a gummy mass turning blue with alkalis. This is formed by the action of the NaHSO, on the O<sub>2</sub>NC<sub>2</sub>H<sub>4</sub>NH<sub>2</sub>, as shown by a check expt., and is probably a hydroquinonedisulfonic acid. From the aminoazo acid and NaHSO, in the autoclave is obtained an acid which has the properties of H,NC,H,-(SO<sub>2</sub>H)<sub>2</sub>OH. Benzeneazosalicylic acid and Na<sub>2</sub>SO<sub>2</sub> give a compound, m. 140°, which is probably PhNHN(SO<sub>2</sub>H)C<sub>6</sub>H<sub>2</sub>(OH)CO<sub>2</sub>H, and H<sub>2</sub>NC<sub>6</sub>H<sub>2</sub>(OH)(CO<sub>2</sub>H)SO<sub>2</sub>H; very little, if any, PhNH, is found. When NaHSO, is used, PhNH, and the aminosulfonic acid are obtained, and the mother liquors evolve NH<sub>2</sub> and turn blue with alkalis. A number of other products are formed, among them a benzidine deriv. m. 170-3°, and a S compd. containing no N and m. 126-8°. Chrysamine and NaHSO, react similarly. C. A. ROUILLER.

Opaque White Printed on Fabric to Produce a Figured Effect. Heilmann & Co., A. Lipp and M. Battegay. Bull. soc. ind. Malhouse, 81, 137-8.—This process is said to be an improvement on the opaque effect produced by printing viscose alone on cotton (Ger. Pat. 70,999, Jan. 13, 1893). It consists in printing a mixt. of viscose, alk. aluminates, and alk. silicates, phosphates, borates, or soaps on the cotton. A subsequent drying or steaming causes a pptn. of cellulose xanthogenate along with insol. Al salts. The fabric is afterward finished in the way customary for viscose without using mineral acids, however (cf. Lilienfeld, C. A., 5, 2737). MILES R. MOFFATT.

The Microscopic Appearance of Bleached and Mercerized Sateen. JULIUS HUBNER. J. Soc. Dyers Colourists, 27, 128-9.—A number of excellent photomicrographs illustrating changes taking place when sateen is bleached and mercerized both with and without tension.

L. A. OLNEY.

Alterations by the Action of Light on Silk Threads Treated with Tin Salts. G. GIANOLI. Chem. Ztg., 34, 105; through Chem. Zentv., 1910, I, 779.—A piece of silk treated with Sn salt, up to 50% of its wt., was exposed to light for 14 days and compared with a similar piece kept in dark. The former yielded 10% of water sol. N substance; the latter 4%. Therefore silk treated with Sn undergoes oxidation and hydrolysis. If O is excluded the silk is not acted upon even in presence of sunlight.

Sidney Born.

The Mercerizing Action of Concentrated Sulfuric Acid and Concentrated Zinc Chloride Solution on Cotton. V. Mineau. Z. Farben. Ind., 9, 65-6; through Chem. Zentr., 1910, I, 1304.—Through 10 min. immersion in H<sub>2</sub>SO<sub>4</sub> (1.53), at 0-1° and subsequent washing, cotton was changed into a transparent leather-like matter which easily takes up water and is much more intensely colored by indigo dyes than ordinary cotton. ZnCl<sub>2</sub> soln. (64° Bé.) did not affect cotton appreciably. Sidney Born.

Condensation Products of Isatin with Oxythionaphthene, Indandione and Indandone (NOELTING, HERZEAUM). 10.

Isomers of Aposafranine (KEHRMANN, et al.). 10.

Reaction between Pigment and Sizing (KOLLMANN). 23.

HOPFIELT: Die Organisation eines Fabrikbetriebes. Leipzig: H. A. Ludwig. 2 M.

Lange, O.: Die Schwefelfarbstoffe, ihre Herstellung und Verwendung. Leipzig: O. Spamer. 26.50 M.

MAYER, K.: Die Farbenmischungslehre und ihre praktische Anwendung. Berlin: 8°, 88 pp., 4 M.

U. S., 999,944, Aug. 8. A. ZART, Vohwinkel, near Elberfeld, and H. SCHWETTZER, Elberfeld, Ger. Assignors to Farben. vorm. F. Bayer & Co., Elberfeld, Ger. Dya, the urea of the azo compd. from m-phenylenediaminesulfonic acid and cresidine, of the formula

is dried and pulverized, Na salt being a dark yellow powder sol. in H<sub>2</sub>O with a yellow and in conc. H<sub>2</sub>SO<sub>4</sub> with a violet color, yielding on reduction with SnCl<sub>2</sub> and HCl m-phenylenediaminosulfonic acid and the urea of aminocresidine which is further decomposed into p-methoxy-o-m-diaminotoluene and CO<sub>2</sub>, dyeing cotton yellow shades fast

to washing after combination with diazotized p-nitroaniline. Formyl-m-phenylenediaminosulfonic acid, 6-nitro-2-aminotoluene-4-sulfonic acid, 4-nitroaniline-2-sulfonic acid, formyl-2,6-diaminotoluene-4-sulfonic acid, formyl-2,4-toluylenediamine-5-sulfonic acid, formyl-p-phenylenediaminosulfonic acid, etc., can be employed, and as 2nd component other amines can be used, e. g., aniline, toluidines, xylidines, acidyldiamines, naphthylamines, etc.

U. S., 1,001,183, Aug. 22. A. ZART, Vohwinkel, and H. SCHWEITZER, Elberfeld, Ger. Assignors to Farben. vorm. F. Bayer & Co., Elberfeld, Ger. Dye, the urea of the azo compd. from 3,5-diaminobenzoic acid and cresidine of the formula

$$\left(\begin{array}{c} NH_{a} \\ CO_{a}H \end{array}\right) = N - \left(\begin{array}{c} OCH_{a} \\ CH_{a} \end{array}\right) + CO$$

being in the form of its Na salt a dark yellow powder sol. in H<sub>2</sub>O with a yellow color and in conc. H<sub>2</sub>SO<sub>4</sub> with a red color, yielding on reduction with HCl and SnCl<sub>2</sub> 3,5-phenylenediaminocarboxylic acid and the urea of aminocresidine which is further decomposed into p-methoxy-o-m-diaminotoluene and CO<sub>2</sub>; dyeing cotton yellow shades fast to washing after combination with diazotized p-nitroaniline. In preparing similar dyes, formyldiaminobenzole acid, 3-nitro-5-aminobenzole acid, or 4-oxalyl-amino-2-aminobenzole acid may be used, as well as other amines as 2nd component, e. g., aniline, toluidines, xylidines, acidyldiamines or naphthylamines.

U. S., 1,001,284, Aug. 22. F. KREISSL and C. SEIBERT, Vienna. Obtaining textil fibers from nettles, hops, sunflower stalks, etc., by treating the material with cold lye for 24 hrs. and then preliminarily b. the material in dil. soda lye, washing, treating in an autoclave boiler with 8% soda lye for 5 min. under 15 atm. pressure, supplying jets of H<sub>2</sub>O to sep. the fibers from any remaining ligneous parts, again b. with 8% soda lye for 3 min. under 10 atm. pressure, applying jets of H<sub>2</sub>O, again b. with 2% soda lye under 3 atm. pressure for 3 min., applying jets of H<sub>2</sub>O, bleaching by electrolysis in NaCl soln., treating for a short time with 1% H<sub>2</sub>SO<sub>4</sub>, washing, drying and treating with glycerol vapor to render the fibers soft and smooth.

U. S., 1,001,286, Aug. 22. A. L. LASKA, Offenbach a/M, Ger. Assignor to Chem. Fabrik Griesheim Elektron, Frankfort a/M, Ger. Disazo dye suitable for the preparation of pigment colors, made by combining the tetrazo compd. of o-dichlorobenzidine (NH<sub>2</sub>: Cl::1:2) with 2 mol. proportions of 1-phenyl-3-methyl-5-pyrazolone, the product being a bright orange-red powder, insol. in H<sub>2</sub>O, sol. in conc. H<sub>2</sub>SO<sub>4</sub> with a red color, yielding on reduction with HCl and SnCl<sub>2</sub> o-dichlorobenzidine and 4-amino-1-phenyl-3-methyl-5-pyrazolone and being readily transformed into a color lake of orange tint fast to oil and H<sub>2</sub>O.

U. S., 1,001,325, Aug. 22. F. ULLMANN, Charlottenburg, Ger. Assignor to A. G. für Anilin Fabr., Berlin. Compound of the anthracene series, having the formula

obtained by acting with phenylhydrazine upon an anthraquinone- $\alpha$ -carboxylic acid, the dry product being a yellowish powder, very difficultly sol. in alc. and ether and sol. in pyridine to a yellow soln. and in conc.  $H_aSO_4$  to a yellow or orange soln. having a weak fluorescence. Similar compds. may be obtained from phenylhydrazinesulfonic acid or bromophenylhydrazine or hydrazine itself, etc., and for the anthraquinone- $\alpha$ -carboxylic acid  $\pi_1$ -chloroanthraquinone-carboxylic acid or  $\pi_2$ -carboxylic acid or the chloride of anthraquinone- $\alpha$ -carboxylic acid, etc., can be substituted.

U. S., 1,001,408, Aug. 22. M. H. ISLER, Mannheim. Ger. Assignor to Bad. Anilin- & Soda-Fabr., Ludwigshafen a/R, Ger. Compound of the anthracene series, obtainable by treating 1,5-dichloro-2,6-dibenzoyldiaminoanthraquinone with a condensing agent, e. g., Na<sub>2</sub>CO<sub>2</sub>, and probably having the formula

a yellow powder, giving a yellow soln in conc. H<sub>2</sub>SO<sub>4</sub>, difficultly sol in ordinary solvents and dyeing cotton yellow. Similar condensation products are obtained from 1-benzoylamino-2-bromoanthraquinone, 1,4-dibenzoylamino-2,3-dichloroanthraquinone, 1-chloro-2-benzoylaminoanthraquinone or 1,3,5,7-tetrabromo-2,6-dibenzoyl-diaminoanthraquinone.

U. S., 1,001,457, Aug. 22. K. SCHIRMACHER, Höchst a/M, and R. LEOPOLD, Hochheim a/M, Ger. Assignors to Farbwerke vorm. M. L. & B., Höchst a/M, Ger. Dye, the 6,6'-diethylthio deriv. of indigo, a greenish black powder, sol. in conc. H<sub>2</sub>SO<sub>4</sub> with a bluish green color, in glacial HOAc with a green color and yielding in alc. or PhNO<sub>2</sub> a dichroistic soln. of a brownish red to green color, readily sol. in alk. hydrosulfite with a yellow color and dyeing cotton and wool green to black tints; made by b. 5-ethylthiophenyl-1-glycine-2-carboxylic acid with acetic anhydride, adding H<sub>2</sub>O after cooling and stirring with NaOH soln. while blowing air into the soln. until the dye is sepd.

U. S., 1,001,458, Aug. 22. 'Idem. Bluish red azo dye; see C. A., 5, 2964.

U. S., 1,001,919, Aug. 29. W. BAUER, Vohwinkel, and A. HERRE and R. MAYER, Elberfeld, Ger. Assignors to Farben. vorm. F. Bayer & Co., Elberfeld, Ger. Dye, a condensation product of dibromoisatin-α-chloride and acenaphthenone, a dark reddish brown cryst. powder, sol. in hot C<sub>2</sub>H<sub>6</sub> with a bluish red and in cold conc. H<sub>2</sub>SO<sub>4</sub> with a green and in hot conc. H<sub>2</sub>SO<sub>4</sub> with a blue color and yielding with hydrosulfite and NaOH soln. a violet vat from which cotton is dyed bluish red. The dye probably has the formula

Br Co Co

Other isatin derivs. may be used, e. g.,  $\alpha$ -isatinanilide, chloro-, bromo-, chlorobromo- or dichloroisatin chloride,  $\rho$ -bromo- $\rho$ -methylisatin anilide,  $\rho$ - methylisatin -  $\alpha$ -  $\rho$ - toluidide, isatin chloride, dibromo -  $\rho$ -naphthisatin chloride, etc.

- U. S., 1,002,026, Aug. 29. F. E. BÖTTIGER, Offenbach a/M, Ger. Assignor to Chem. Fabrik Greisheim Elektron, Frankfort a/M, Ger. Giving the scroop of silk to mercerized cotton goods dyed with sulfurized dyes, by scouring the goods, after passage through a soap bath, with a soln. containing tartaric or lactic acid 1 and Na tartrate or lactate 3-5 parts.
- U. S., 1,002,066, Aug. 29. A. LÜTTRINGHAUS, Ludwigshafen-on-the-Rhine, Ger. Assignor to Badische Anilin- & Soda-Fabr., same place. Dye, obtainable from 1-haloanthraquinone-2-carboxylic acid and p-chloroaniline and having the formula

being when dry a carmin-red powder which yields a red soln. in hot amyl acetate, insol. in 65% H<sub>2</sub>SO<sub>4</sub> but giving a reddish yellow soln. in 70% H<sub>2</sub>SO<sub>4</sub>, and dyeing cotton bluish red.

- U. S., 1,002,118, Aug. 29. M. BECKE, Höchst a/M, Ger. Assignor to Farbwerke vorm. M. L. & B., same place. Producing multicolored effects in spun and woven goods by pptg. on vegetable fibers dyed with dyes resisting oxidizing agents, Mn oxides, then working the fibers thus treated together with untreated fibers, dyeing the goods thus made, and subsequently treating the goods with agents for developing the oxidizing action of the Mn oxides.
- U. S., 1,002,270, Sept. 5. M. HESSENLAND, Höchst a/M, Ger. Assignor to Farbwerke vorm. M. L. & B., same place. Dye, 4-aminoanthraquinone- $\alpha$ -acridone containing the radical  $\alpha$ -anthraquinonyl in the amino group, a black powder insol. in  $H_2O$ , alc., ether, alkalies and dil. mineral acids, sol. in conc.  $H_2SO_4$  with an olivegreen color and in alk. "hydrosulfite" with a reddish brown color and dyeing from this soln. cotton gray to black tints; made by reacting on 4-aminoanthraquinone- $\alpha$ -acridone with  $\alpha$ -chloroanthraquinone. The following dyes may be similarly obtained from 4-aminoanthraquinone- $\alpha$ -acridone:  $+\beta$ -Chloroanthraquinone, dark green, olive-green (in  $H_2SO_4$ ), reddish brown (color of vat), grayish green (dyeings on cotton); + benzoyl-chloride, blue, reddish brown, violet, reddish blue; + bromobenzene, dark green, brown, violet, green; +  $\beta$ -chloronaphthalene, blue, grayish brown, reddish brown, bluish gray; +  $\beta$ -chloronaphthalene, dark green, brown, violet, yellowish green.
- U. S., 1,002,408, Sept. 5. E. KNOEVENAGEL, Heidelberg, Ger. Dyeing acetyl cellulose after preliminary treatment with an aq. soln. of an aromatic amine, e. g., aniline, p-nitroaniline,  $\alpha$ -naphthylamine, aminoazobenzene, benzidine, or p-aminophenol, to constitute one component of a dye to be formed in the material and subsequent rinsing before treatment with other dye-forming component or diasotizing.

- U. S., 1,002,635, Sept. 5. W. Bratkowski, Berlin, Ger. Apparatus for measuring and regulating the concentration of dye-liquors, the supply of fresh dye-liquor being regulated electrically by a Se cell acted on by a const. source of light passing through a body of the dye-liquor circulating between glass plates.
- U. S., 1,002,825, Sept. 12. C. DE LA HARPE and E. BODMER, Basel, Switzerland. Assignors to Dye-Works formerly L. Durand, Huguenin & Co., same place. Dyes, resulting from the condensation of a resorcinolsulfonic acid with an amino-m-oxybenzoylbenzoic acid substituted in the amino group, e. g., dimethylamino-m-oxybenzoylbenzoic acid being brown-red to red powders easily sol. in H<sub>2</sub>O in the form of their alkali salts to solns. colored from red-orange to raspberry-red and showing a green fluor-escence, from which they are pptd. by mineral acids, sol. in conc. H<sub>2</sub>SO<sub>4</sub> with a citron yellow to a brown-red color, turning to rose or yellow-orange on addition of H<sub>2</sub>O, and dyeing unmordanted wool and chromed wool or cotton orange to magenta red fast to Cl and light.
- U. S., 1,003,257, Sept. 12. C. DE LA HARPE and E. BODHER, Basel, Switzerland. Assignors to Dye-Works formerly L. Durand, Huguenin & Co., same place. Dyes made by condensation of a pyrogallolsulfonic acid with an amino-m-oxybenzoylbenzoic acid substituted in the amino group, e. g., with diethylamino-m-oxybenzoylbenzoic acid, the products being brown-red to brown powders easily sol. in H<sub>2</sub>O in the form of their alkali salts to solns. colored from scarlet to bluish red, from which they are pptd. by mineral acids, sol. in conc. H<sub>2</sub>SO<sub>4</sub> with a red color turning to rose-yellowish to yellow-orange on addition of H<sub>2</sub>O, and dyeing chromed wool or cotton violet to brown-violet and unmordanted wool red-violet to violet, fast to light, fulling and S.
- U. S., 1,003,266, Sept. 12. H. JAESSCHIN, Berlin and O. KALTWASSER, Dessau, Ger. Assignors to A. G. für Anilin Fabr., Berlin. Monazo dye adapted for the production of violet lakes and dyeing wool from an acid bath after chroming on the fiber violet shades; made by combining diazotized 4-chloroaminophenol-6-sulfonic acid with 2-naphthol-3-carboxylic acid, the Na salt of the dye being a bluish black powder, sol. in H<sub>2</sub>O to a claret soln. unaltered by the addition of Na<sub>2</sub>CO<sub>2</sub> but assuming a more reddish color on the addition of NaOH or an acid, sol. in conc. H<sub>2</sub>SO<sub>4</sub> to a bluish red soln., from which on the addition of ice red-brown flakes of the dye are sepd. and yielding with strong reducing agents 4-chloro-2-aminophenol-6-sulfonic acid and 1-amino-2-naphthol-3-carboxylic acid.
- U. S., 1,003,268, Sept. 12. R. Just and H. Wolff, Ludwigshafen-on-the-Rhine, Ger. Assignors to Badische Anilin- & Soda-Fabr., same place. Dye, dichloroiso-violanthrene, made by treating isoviolanthrene with SO<sub>2</sub>Cl<sub>2</sub>, a dark violet powder insol. in H<sub>2</sub>O and in dil. acids and alkalies, sol. in conc. H<sub>2</sub>SO<sub>4</sub> to a green soln., sol. in PhNO<sub>2</sub> with a magenta-red color and sol. in alkaline hydrosulfite soln. to a blue vat which dyes cotton blue, changing on washing and drying to very fast reddish violet. Brominated isoviolanthrene has similar properties.
- U. S., 1,003,293, Sept. 12. H. POLIKIER, Leipzig, Ger. Assignor to A. G. für Anilin Fabr., Berlin, Ger. Monoazo dye derived from diazotized 2,5-dichloroaniline and ethylbenzylanilinesulfonic acid, its Na salt being an orange powder, easily sol. in H<sub>2</sub>O to a yellow soln. which is unaltered by alkali but changed to red by mineral acid, sol. in conc. H<sub>2</sub>SO<sub>4</sub> with a yellow color, turning to red on the addition of ice with sepn. of the free dye, yielding on reduction 2,5-dichloroaniline and 4-aminoethyl-benzylaniline sulfonic acid, and dyeing wool from an acid bath yellow.
- Brit., 9,359, Apr. 18, 1910. FARBEN. VORM. F. BAYER & Co., Elberfeld, Ger. Mfg. halogenated vat dyes contg. no more than 3 atms. of halogen, by condensing

 $\alpha$ -keto derivs. of isatin, or of its homologs or its halogen substitution products (in which the O of the  $\alpha$ -keto group is replaced by easily movable or replaceable substituents,  $\epsilon$ . g., halogen S, the amino group, the alkoxy group) with 1-naphthol or with  $\rho$ -monohalogen substituted derivs. thereof and treating the soln. of the resulting condensation products in conc.  $H_{\alpha}SO_{\alpha}$  with Br

Brit., 15,070, Dec. 21, 1910. HERBERT LEVINSTEIN, Crumpsall Vale, Blackley, Manchester. Mfg. intermediate products for the manufacture of azo dyes by condensing naphthylaminosulfonic acid or heteronuclear aminonaphtholsulfonic acid with a giveerolchloro- or giveerobromohydrin.

Brit., 16,629, July 12, 1910. R. PAWLIKOWSKI, Luisenstrasse 13, Gorlitz, Ger. Treatment of artificial threads; see C. A., 5, 3169.

Brit., 19,545, Aug. 2, 1910. FARBEN. VORM. F. BAYER & Co., Elberfeld, Ger. Mfg. azo dyes by combining 1 or 2 mols. of diazotized 2-aminobenzoylamino-5-naphthol-7-sulfonic acids or 2-sulfaminobenzoylamino-5-naphthol-7-sulfonic acids or of their substitution products or 2 mols. of a mixt. of these compds. (or 1 mol. of any of these compds. + 1 mol. of a diazo compd., such as diazo-p-nitroaniline) with 1 mol. of the sym. urea of 2,5,7-aminonaphtholsulfonic acid or with 1 mol. of 5,5'-dihydroxy-2,2'-dinaphthylamine-7,7'-disulfonic acid.

Brit., 21,200, Sept. 12, 1910. Idem. Mfg. an azo dye by combining p,p'-diamino-diphenylurea-m,m'-disulfonic acid with 2 mols. of m-phenylenediamine.

Brit., 21,202, Sept. 12, 1910. Idem. Mfg. azo dyes by producing p-aminoazo compds. by combining with aromatic amines of the benzene series, such as o- or m-toluidine, p-xylidine, cresidine, etc., diazotized aminonaphtholsulfonic acids or diazotized derivs. thereof (such as for instance aminobenzoylaminonaphtholsulfonic acids or aminophenyloxynaphthimidazolesulfonic acids, etc.) which derivs. are substituted in the amino group by substituents contg. a heteronuclear amino group capable of being diazotized, and in converting the aminoazo compds. thus obtained into the sym. diarylurea or diarylthiourea compds. by treatment with phosgene or thiophosgene or their equivs., such as perchloromethylformate or hexachlorodimethylcarbonate.

Fr., 402,546, Apr. 30, 1909. *Idem*. In the production of azo dyes, employing the halogen derives of different aminophenyl ethers or their homologs.

Ger., 236,592, Dec. 18, 1908. Addition to 232,778, Dec. 1, 1908; cf. C. A., 5, 1522. FARBEN. VORM. F. BAYER & Co., Elberfeld. Production of fast dyeings. In the principal patent the reduction products of alizarin reduced to the anthranol state (wherein both ketone groups are reduced) are shown to be valuable dyes on chrome mordants. The products may be advantageously dyed out according to the after-chroming or single bath dyeing process. Examples are given for the application of desoxyalizarin, desoxyanthrapurpurin, and desoxyflavopurpurin.

Ger., 236,656, Dec. 22, 1910. CHEM. FABRIK. VORM. SANDOZ, Basel. Manuf. of a monobromonaphthalene-1-diazo-2-oxide-4-sulfonic acid. The stable anhydride-like diazo compds. of o-aminooxynaphthalenes may be directly brominated, with the exit of H<sub>2</sub>O, under suitable conditions, to bromonaphthalenediazo oxides. The monobromonaphthalene-1-diazo-2-oxide-4-sulfonic acid is especially valuable for the production of azo dyes. It can be obtained with a good yield by treating with Br, at a raised temp., naphthalene-1-diazo-2-oxide-4-sulfonic acid, dissolved in chlorosulfonic acid or conc. H<sub>2</sub>SO<sub>4</sub>—in the latter case preferably in the presence of a halogen carrier. The product is purified by dissolving it in H<sub>2</sub>O at 75°, filtering, and pptg. with conc. HCl. Properties are specified.

## 26. PIGMENTS, RESINS, VARNISHES AND INDIA RUBBER.

### A. H. SABIN, THEODORE WHITTELSEY.

Action of Light on Pigments. III. A. EIBNER. Chem. Ztg., 35, 786; cf. C. A., 5, 3628.—Not only does ZnO act as previously described (to destroy color), but so does ZnCO<sub>2</sub>, ZnSiO<sub>3</sub>, and ZnS, in a less degree. The effect on Fe pigments is less noticeable because of their dark color. Colors pptd. on chrome-red are not resistant to ZnO treatment. But in general colors pptd. with mordants, such as tannin, etc., appear more permanent than those on an inactive base. A new pigment called "limegreen" (Kalkgrüne) appears quite fast to light. The literature of the subject and of photochemistry is discussed in considerable detail.

A. H. Sabin.

A New Method of Freeco Painting. G. Leuchs. Chem. Ztg., 35, 821.—A foundation coat is made of a mixt. of water-slaked lime and brick dust or dolomite sand; this is non-absorbent, and is essential. The surface coat 1.5-2 mm. thick is a mixt. of Ca(OH)<sub>2</sub> with marble dust, mixed with some powdered zeolite, pumice, or trass, and containing 1.5% plaster of Paris. On this absorbent coating, which is kept moist, the painting is done; if by action of CO<sub>2</sub> it becomes non-absorbent it is washed with dil. H<sub>2</sub>SO<sub>4</sub>. Finally it is repeatedly sprayed with Ba(OH)<sub>2</sub> soln. avoiding the formation of drops on the surface; if any, they are removed with a dry cloth. This forms BaSO<sub>4</sub> and Ca(OH)<sub>2</sub> which latter changes to CaCO<sub>2</sub>; the surface is hard, non-porous, non-absorbent and permanent.

A. H. Sabin.

Effect of Pigments Ground in Linseed Oil. H. A. GARDNER. J. Ind. Eng. Chem., 3, 628.—Five g. each of oil (acid no. 1.84) and pigment were ground in mortar and stood 1 month, the oil then extracted with benzine, evapd., burned and ignited. The amt. of pigment dissolved was negligible except ZnO and white lead about 0.1 and Pb<sub>2</sub>O<sub>4</sub> 0.2%. Insol. pigments have no chem. action. Pb and Zn pigments saponify oil; this may be prevented by adding "a moderate %" of inert pigment, which latter exert only contact action. Note on same. A. H. Sabin, 1bid., 3, 790.—Insol. bodies which show contact action are not chemically inert. S. denies that white lead and ZnO saponify oil, or the oil would be more completely saponified in presence of excess of pigment; also that the presence of a small amt. of a chemically inactive insol. substance can prevent saponification.

A. H. Sabin.

Analysis of Mixed Paints. E. F. Ladd. N. Dak. Agr. Coll. Expt. Sta. Point Bull. 1, No. 5.—This is a report of 22 analyses of paints.

S. C. Clark.

Report on Service Condition of Paints. N. Dak. Agr. Coll. Expt. Sta. Paints
Bulls. 1-4.—These bulletins contain the reports of 4 committees, on the conditions
of the various paints on the panels of an experimental fence. The details of the expts.
are given and the comp. of the paints.

S. C. Clark.

Synthetic Rubber. Frank E. Barrows. The Armour Engineer, 3, 197; Chem. Eng., 14, 355-62.—An authoritative review.

D. Spence.

The Theory of Vulcanization of Caoutchouc. F. W. HINRICHSEN. Z. Chem. Ind. Kolloide, 8, 245.—H. objects to the theory advanced by Wo. Ostwald (C. A., 5, 3170) and points out the difficulties experienced in trying to reverse the adsorption process of S (Alexander, C. A., 5, 1525). These difficulties are of chem. rather than physical origin. Combined S passes quantitatively into the derivs. of rubber. Why should not S and S<sub>2</sub>Cl<sub>2</sub> combine with caoutchouc in the same manner as Br, Cl, HCl, ozone, etc. H. thinks that, owing to the different rate of reaction, at first a surface reaction takes place which is then followed by a chem. combination of the adsorbed substances. This would mean that in time the free S decreases while the combined S increases, a process which would explain the so-called "after-vulcanization." A sample

of vulcanized rubber exposed to ordinary temp. for 6 months showed a decrease in free S from 4.5-1.1% and an increase in the combined S from 4.5-7.5%. At higher temps, these same results were obtained within a few days. The total S was detd, by electrolytic oxidation with HNO<sub>3</sub>, and then gravimetrically or volumetrically with benzidine according to Raschig; the free S was detd, by extracting 10 hrs. with acctone, then oxidizing with HNO<sub>3</sub>, b. down with HCl, adding NaCl and then pptg. as BaSO<sub>4</sub>. By following the above method the adsorption of S proceeds in such a manner that it can be easily followed and measured; at the same time it affords an opportunity of studying the influence of filling materials and of higher pressure on the rate of vulcanization.

Some Constituents of Parthenium argentatum Gray, the Source of Guayule Rubber. P. ALEXANDER. Ber., 44, 2320-8.—A. has investigated the acetone-sol. constituents and ethereal oil derived from the guayule plant. The acetone-sol. constituents were obtained by the direct extraction of the plant (yield 6.5% of original material). Of the total ext. 54% dissolved in petroleum-ether, 31% in ether and the rest in 90% alc. The petroleum-ether ext. was soft and sticky whereas the ether and particularly the alc. exts. were much harder. The petroleum-ether ext. contained only 12.1% of unsapon. matter, the ether ext. 7.0% and the alc. ext. 2%. Small quantities of an acid, m. 119°, were obtained from the alk. liquors. This acid readily changes and A. believes that it belongs to the cinnamic series; he finds additional support for this view from the fact that from the unchanged resin from guayule rubber obtained by the alkali process, a considerable quantity of a cryst. acid needles from ether m. 79° of the formula CaHaOa was obtained which is probably phenylacetic acid (little experimental evidence; even analysis shows a wide disagreement with theory.—ABSTR.). Ethereal oil: This was obtained by steam distillation of the wood (11/2-4% yield) as a greenish yellow oil with a peculiar odor of pepper. Repeated fractional distillation gave the following: Fraction I  $b_{17}$  57-8°,  $b_{780}$  155-7°,  $d_{18}$  0.8602,  $\alpha_{D}^{16}$  -3° 22′ (in 100 mm. tube),  $n_D^{16}$  1.478, fraction II  $b_{17}$  130-40°,  $d_{16}$  0.9349,  $\alpha_D^{16}$  -21° 24′,  $n_D^{16}$  1.496. That fraction I was pinene was confirmed by prep. of the nitrosochloride (m. 100-2°), nitrolbenzylamine (m. 122°) nitrolpiperylamine (m. 128°) derivs. Analyses of fraction II agree with the formula (C,H,),. This substance has an intensive ador of pepper and gives guayule rubber its characteristic ador. A. believes it is a sesquiterpene, C<sub>11</sub>H<sub>24</sub>, but efforts to identify it with known sesquiterpenes failed. In the steam distillation of the acetone ext. of the wood, besides the ethereal oil, a solid cryst. substance (m. 127-8°) with pleasant aromatic camphor-like ador was obtained. Its formula is apparently C<sub>18</sub>H<sub>24</sub>O and is probably a sesquiterpene alc. Samples of the ethereal oil obtained from shrub which had lain exposed to air for a long period contained none of the higher b. fraction, this having been converted by oxidation into resinous products. Styrene could not be detected in the first fractions from the distillation of the ethereal oil. The difficulties encountered in the vulcanization of guayule rubber in the early days A. attributes to the presence of these ethereal oils in the rubber. D. Spence.

Effect of Alcohol Denatured with Wood Spirit on Health (HORBACZEWSKI). 22.

Para Rubber Seed as a Source of Oil. 27.

Elaidin Reaction (FOKIN). 27.

Papers of the 1909/1910 Paint and Varnish Society. Edited by the Secretary. London: B. Blackler.

U. S., 1,001,222, Aug. 22. F. RICHTER, Frankfort a/M, Ger. Manufacturing carbon of high clarifying activity by subjecting to dry distillation finely divided C used

- for clarifying and containing mineral matter, e. g., Fe compds., which will not frit together when heated to the distillation temp. and charged with organic matter from the clarification, so as to transform the latter into active C.
- U. S., 1,001,415, Aug. 22. J. KOETSCHET and J. C. A. MEYER, Lyon, France. Making zinc sulfide by pptn. from an acid bath and maintaining the pptd. sulfide for a long time (preferably 14 days at ordinary temp. or 24-36 hrs. at 80°) in contact with dil. acid, e. g., 2% H<sub>2</sub>SO<sub>4</sub>, to obtain a product which when used as a pigment will not turn yellow or blacken in the air.
- U. S., 1,001,585, Aug. 22. J. F. GLIDDEN, Cleveland, Ohio. Decorating surfaces of steel, wood, glass, paper, etc., by applying a coagulable oil, e. g., Chinese wood oil, with a pigment if desired, to the surface and subjecting the oil to acid fumes, e. g., HNO<sub>2</sub>, to cause coagulation and hardening and give the surface a cellular appearance.
- U. S., 1,002,246, Sept. 5. C. ELLIS, Montclair, N. J. Making white lead by raising Pb to a temp. about 400° above its m. p., comminuting by a heated jet of air under pressure so that filamentous Pb coated with a catalytic oxide is produced and subjecting a continuously, integrally and progressively advancing stream of the product to a gaseous current containing O under substantially atm. pressure in the presence of moisture to cause hydration and converting the product into basic carbonate of Pb by the prolonged action of O, CO, and moisture at a temp. above that of the atm.
- U. S., 1,002,380, Sept. 5. E. Eusron, St. Louis, Mo. Converting light, fluffy, over-carbonated white lead (3PbCO<sub>2</sub>.PbO<sub>2</sub>H<sub>2</sub>) into commercial white lead of the formula 2PbCO<sub>2</sub>.PbO<sub>2</sub>H<sub>2</sub>, by treating it with a soln. of neutral Pb(OAc)<sub>2</sub> of acid reaction and agitating.
- U. S., 1,002,644, Sept. 5. H. Z. COBB, Winchester, Mass. Assignor to Revere Rubber Co., Boston, Mass. Making rubber hose, etc., by forming a tubular textil web while maintaining around it a mass of plastic dough-like rubber comp. through which the yarns pass on their way to form the web, so that they are impregnated and the web is coated with the material.
- U. S., 1,002,667, Sept. 5. T. GARE, New Brighton, England. Apparatus for reforming rubber articles from powdered waste rubber.
- U. S., 1,003,244, Sept. 12. W. ELSNER and O. MEYER, Hanover, Ger. Purliying rubber or gutta-percha by extracting the resins and H<sub>2</sub>O-sol. impurities by treating with acetone oils and adding H<sub>2</sub>O to the oils, then sepg. the H<sub>2</sub>O soln. of the extracting liquid from the acetone oils containing the resins and recovering the oils from the resins.
- U. S., 1,003,438, Sept. 19, W. F. Doerflinger, Brooklyn, N. Y. Making a solution for use as a lacquer, varnish, etc., by dissolving acetyl cellulose in diacetone alc., with or without nitrocellulose.
- U. S., 1,003,741, Sept. 19. A. HESSE, Wilmersdorf, Ger. Making solutions of resins, e. g., copal resin, by heating with phthalic acid esters, e. g., the di-Me or di-Et ester.
- Brit., 9,219, Apr. 16, 1910. WM. R. HODGKINSON, 89 Shooters Hill Road, Black-heath, S. E. The condensation or polymerization of isoprene or mixts, contg. isoprene, to form rubber, or a substance resembling rubber, by heating the isoprene or mixt. with an amide of an alkali metal (such as NaNH<sub>2</sub>), or a nitride of the type of Mg<sub>8</sub>N<sub>2</sub> in the absence of an oxidizing agent.
- Brit., 16,407, July 9, 1910. R. BRIDGE, Castleton Iron Works, Castleton. Treating rubber vines, shrubs, bulbs, or the like for the sepn. of the rubber and barky matter, by first subjecting them while dry to the crushing action of 1 or a plurality of pairs of

rollers, or to I or a plurality of pairs of reciprocable flat plates, removing the sepd. woody matter by a current of air, and then passing them I or more times in a wet state through other crushing rollers arranged to give a plurality of nips while being simultaneously treated to a shower of hot or cold H<sub>2</sub>O or H<sub>2</sub>O'and steam.

Brit., 17,770, July 26, 1910. A. S. RAMAGE, 1543 Niagara St., Buffalo, N. Y. In the prep. of a pigment, replacing a part at least of the O or OH constituent of a basic Pb compd. with a satd. organic acid, e. g., a higher fatty acid such as stearic acid.

Brit., 21,401, Sept. 14, 1910. F. POLLAK, Eislebenerstrasse 14, Berlin W., Ger. Mfg. compact, insol., opaque, white condensation products from phenols and formal-dehyde, by treating a substance of the phenolic series with HCHO in the presence of so small a quantity of acid that the latter shall not exceed 1.5% of the insol. final product, and coagulating and hardening the intermediate product by the action of heat.

Brit., 28,329, Dec. 6, 1910. P. HAMN-NIEDERLAHNSTEIN, Rhineland, Prussia. Mercerizing cotton in the form of skeins by stretching the material, introducing it completely immersed in the lye, slackening while submerged and again stretching after it has shrunk sufficiently, and subsequently submitting it to the known finishing process.

Brit., 29,389, Dec. 17, 1910. A. RÖTER, Brunnenstrasse 32, Pyrmont, Ger. A quick drying ink consisting of ordinary ink combined with a very volatil neutral substance, such as conc. spirits of wine or other analogous substances.

Brit., 29,997, Dec. 24, 1910. E. Scheitlin, 23 Austrasse, Basel, Switz. Rendering alkali superoxides harmless by combining them with resins. This product is gelatinized and molded. Further details are specified.

# 27. FATS, FATTY OILS AND SOAPS.

#### E. SCHERUBEL.

Examination of the Stearin of Wool Fat. E. Coen. Soc. chim. ital., Feb. 22, 1911; through J. Soc. Chim. Ind., 30, 755.—Wool fat contains 18-28 % unsaponifiable matter. Its sapon. no. is 118-136 (the mean value 163 for the fat itself is lower than that of sterin from other fatty substances). The acetyl no. of the unsaponifiable portion is 23-37%; higher alcs. calc. as cholesterol are 18.4-27.3%. The hydrocarbons are solid, having a d. 0.924-0.936 at  $15^{\circ}$ , I no. 26-34, optical rotation at  $20-25^{\circ}$ ,  $+11.7^{\circ}$  to  $+15.2^{\circ}$ . E. J. WITZEMANN.

The Elaidin Reaction. Preliminary Communication. S. FOKIN. J. Russ. Phys. Chem. Soc., 42, 1068-73.—The supposition is made that only such acids are capable of giving the elaidin reaction as, like H<sub>2</sub>SO<sub>2</sub> and HNO<sub>2</sub>, exist in 2 modifications. This is corroborated by showing that H<sub>2</sub>PO<sub>2</sub> also gives this reaction. The product obtained by warming pleic acid with H<sub>2</sub>PO<sub>2</sub> to 170-80° in an atm. of H or in sealed tube m. 28-9°. A similar reaction takes place with H<sub>2</sub>PO<sub>2</sub>, PCl<sub>2</sub>, EtNO<sub>2</sub> and C(NO<sub>2</sub>)<sub>4</sub>. Whether the elaidin reaction is reversible could not be detd. On heating pure elaidinic acid to 180° with H<sub>2</sub>PO<sub>3</sub> for 30-40 hrs. a liquid acid is obtained which seems to be a third modification of oleic acid. This modification does not give the elaidin reaction with H<sub>2</sub>PO<sub>3</sub>, and upon reduction with H + Pt is quant. transformed into stearic acid.

Retimation of Small Quantities of Free Acids. H. Lorbell. Seifensieder Ztg., 1911, 501-2, 530-2; Petroleum, 6, 2074, 2182-3; see C. A., 5, 2341.

O. E. BRANSKY.

Utilization of Para Rubber Seed as a Source of Oil. Anon. Bull. Imp. Inst., 9, 35-8; through J. Soc. Chem. Ind., 30, 755.—Owing to the rapid increase in the productive area of Para rubber plantations, the use of the seeds for oil is likely to become important. Precautions must be taken in the extraction of the oil to minimize the action of the lipolytic enzyme present in the seeds. Moreover, as the seeds contain a cyanogenetic glucoside and an enzyme capable of decomposing it, extensive feeding tests are necessary to ascertain whether the residual cake is of value as a cattle food.

E. Scherubert.

Cultivation, Production, Preparation and Utilization of Castor Seed. Anon. Bull. Imp. Inst., 9, 17-35; J. Soc. Chem. Ind., 30, 754-5.—A general article describing the cultivation of the plant and the prep. of the seeds and oil. The decorticated seeds are ground with H<sub>2</sub>O to coagulate protein matter. The solid matter is strained off, dried and pressed to secure the oil. The meal prepared by this process contains H<sub>2</sub>O 9.2%, ash 7.3%, oil 2.6%, protein 71.7%, crude fiber 5.0%, and carbohydrates 4.2%.

Determination of the Density of Oils. E. Beilmer. Chem. Zig., 35, 997.—A method employing the well known alc.-H<sub>2</sub>O mixt. carried out as follows: To 2 cc. of abs. alc. H<sub>2</sub>O is added until drops of oil, introduced at the start, just begin to float. The d. of the mixt. is then detd. A table is given showing d<sub>10</sub> for mixts. containing from 0.1 cc. H<sub>2</sub>O up to 4 cc.

E. SCHERUBEL.

Analysis of Cottonseed Products. F. Paquin, E. L. Johnson, E. R. Barrow, F. N. Smalley and R. C. Warren. Oil Paint and Drug Rep., June 12, 1911; J. Soc. Chem. Ind., 30, 906.—Well known methods with slight modifications. E. S.

The Examination of Oils. H. DUPER. Mitt. Lebenson Hyg., 2, 65-79; through J. Soc. Chem. Ind., 30, 907.—For detg. the critical temp. of soln., D. recommends a mixt. of 2 solvents, in one of which (isobutyl alc., amyl alc., acetone, petroleum ether or aniline) the oil is readily sol. and in the other (EtOH) less readily sol. Using a mixt. of 1 vol. of anilne and 4 vols. of alc. the following values were obtained: Olive oil (6 samples), 35.7-41.2°; arachis oil (3), 42.6-43.3°; sesame oil (2), 30-33.4°; poppy-seed oil (1), 12.1°; walnut oil (4), 29.6-33.1°; colza oil (1), 42.3; cottonseed oil (1), 27.2°. The vol. of an oil does not increase proportionally with the temp. For ordinary purposes, the  $d_{18}$  can be calc. from that at higher temps. by means of the equation: dt = dt' + 0.000665 (t' - t). As the diff. (t' - t) decreases, the deviation of the calc. from the observed d. increases. For t' = 98 the  $d_{18}$  can be calc. to the third decimal place by means of the formula  $d_{18} = d$  at  $98^{\circ} + 0.0552$ .

Peculiar Adulteration of Wax. H. MALFATTI. Z. anal. Chem., 50, 693-4.—Upon exam. of a wax of A. D. 1430, a peculiar and strong odor was noted such as is characteristic of genuin fresh waxes. This was found to be a product of the umbelliferae, Asafoetida.

E. SCHERUBEL.

Determination of the Cleansing Power of Soaps. A. A. Zhukov and P. I. Shestakov. Chem. Ztg., 35, 1027.—The difficulty in detg. the cleansing power of soaps is due chiefly to the difficulty of obtaining homogenously dirty clothes. To obviate this a soln. of lanolin and lampblack in benzine was made and cotton goods immersed in same. By washing cloth thus treated with different soaps in a washing machine, with proper control of time, temp. and conc. of soap soln. comparative results may be obtained. It was found that the best soaps for cleansing arrange themselves as follows: (1) Tallow soap, (2) soaps made from liquid vegetable oils and olein, (3) coconut and palm kernel oil soaps, and (4) rosin soaps. The conc. of the soap soln. exerts a great influence upon the detergent power; the best results are obtained with a 0.2-0.4% soln.

Manufacture of Pure Animal Charcoal with Recovery of By-products. J. PAR-DELLER. Seifensieder Ztg., 38, 953-4, 989-90, 1013-4, 1037-8, 1072-4. E. S.

Cotton Wax (KNECHT, ALLAN). 25.

Analysis of Ceresin and Paraffin Mixtures (CHERCHEFFSKY). 22.

Determination of Fats in Feeding Stuffs (SCHULZE, et al.). 12.

Apparatus for Melting Point of Fats (LIEBEMANN). 1.

COWAN, T. W.: La cire. Paris: 8°, 200 pp.

U. S., 1,002,186, Aug. 29. A. SOLOMONOFF, Syracuse, N. Y. Combining ammonia with fatty acids to produce a harder and amorphous material by treating the fatty acids, e. g., stearic or palmitic, in a granular or powdered condition with anhydrous NH<sub>2</sub> or other alkali.

U. S., 1,003,259, Sept. 12. C. Hill, San Antonio, Tex. Apparatus for extracting wax from plants.

## 28. SUGAR, STARCH AND GUMS.

### A. HUGH BRYAN.

The Sugar Beet Industry in Nevada. C. S. KNIGHT. Univ. of Nev. Agr. Expt. Sta. Bull. 75.—Includes a report of 63 analyses by S. C. Dinsmore. S. C. CLARK.

The Formation of Sugars and Starch in the Sweet Potato. T. E. Keitt. Clemson Coll. S. C. Agr. Expt. Sta. Bull. 156.—While definit conclusions cannot be drawn as yet, it is stated that the character of the soil and the meteorological conditions for a few days preceding digging affect the content of the potatoes. The highest % of starch was found in the tubers just after the first killing frost.

S. C. CLARK.

The Sugar Contained in the Bulbs of Nephrolepis hirsutula Presl. G. D. LIEBLER. Univ. Innsbruck. Ber. bot. Ges., 29, 375-80.—The 50% alc. ext. of the dried material showed properties which indicate the presence of d-glucose and d-fructose.

H. A. SPORHR.

Simultaneous Coagulation of Two Colloids (TIEBACEX). 2. Molasses Fodders (CROCHETTELLE, MILON). 12. Sugars and Starch in the Sweet Potato (KEITT). 12. Enzymes of Diastase (LYATIN). 11.

## 20. LEATHER AND GLUE.

#### WALTER J. KIETH.

Constitution of Tannic Acid. M. NIERENSTEIN. J. Soc. Dyers Colourists, Feb., 1911; J. Am. Leather Chem. Assoc., 6, 352-62.—A concise history of the researches on tannic acid from 1787-1911. N. concludes that tannic acid is a mixt. of at least 2 compds., digallic acid and leucotannic acid.

CHAS. R. OBERFELL.

Simultaneous Coagulation of Two Colloids (TIEBACEE ). 2. Detection of Wood Pulp Liquor in Tanning Extracts. 23.

BENNET, H. G.: The Manufacture of Leather. New York: Van Nostrand. 441 pp., \$4.50.

- U. S., 1,003,124, Sept. 12. O. RÖHM, Darmstadt, Ger. Bating hides and skins by subjecting them to the action of enzymes of the pancreas and of trypsin of vegetable origin in a fermenting or putrefying liquor containing salts of NH<sub>4</sub> or other alkali.
- U. S., 1,003,205, Sept. 12. D. REDDAN, Fitzroy, Victoria, Australia. Treating hides, in the manufacture of leather, first removing the hair from the hide, then treating it with a soln. formed from alum 9 oz., "arsenic" 8 oz., soft soap 4.5 lbs., and NaOH 1 lb. per gal. of H<sub>2</sub>O, then coating and working into the pores of the hide a rubber dough comp. consisting of equal amts. of ordinary rubber, reclaimed rubber, a mixt. of pulverized asbestos and ZnO, a mixt. of S, paraffin, beeswax and tallow, lampblack, and a mixt. of PbO and S and then finally subjecting the hide to a curing treatment, by the use of heat.

Brit., 17,596, July 25, 1910. F. G. Browne, Dandenong Road, Murrumbeena, Victoria, Australia. In a prep. for treating skins, or leather, the employment therein of cut up or pulverized old vulcanized rubber from which the S has been extracted by means of Na<sub>2</sub>SO<sub>2</sub>.

### 30. PATENTS.

Abstracts of patents are included under the foregoing divisions of the Journal. The abstracts of U. S. Patents are prepared by Earl T. Ragan, those of the British, French and German Patents by O. D. Swett and those of Canadian Patents by Russell S. Smart, of Fetherstonhaugh & Co., Ottawa (5 Elgin St.). The abstracts of French Patents are prepared, with permission, on the basis of the abstracts in Moniteur scientifique, those of German Patents, with permission, on the basis of the abstracts in Chemisches Zentralblatt.

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